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Proceedings of the

FOURTH
COTTON UTILIZATION RESEARCH CONFERENCE

Held at New Orleans, Louisiana
April 30 and May 1, 1964

Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

PREFACE

The Cotton Utilization Research Conference is sponsored annually by the Southern Utilization Research and Development Division to bring the results of recent studies in the areas of mechanical processing, chemical modification, and fundamental research on cotton, to the attention of both executive and research personnel in the textile and allied industries.

This Conference was held April 30 and May 1, 1964, at the Sheraton-Charles Hotel in New Orleans, Louisiana. Serving as Honorary Chairman was Otto Goedecke, of Otto Goedecke, Inc., and as General Chairman, George S. Buck, Jr., of the National Cotton Council of America. The program was developed by B. H. Wojcik, Assistant Director, under the guidance of C. H. Fisher, Director, in cooperation with staff members, and advisers representing the cotton industry.

These proceedings report in full or summary the statements presented by the various speakers during the conference and give an account of the discussions following.

Numbers in parentheses refer to references or literature cited at the end of the article. The figures and tables are reproduced essentially as they were supplied by the writer of each paper.

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WELCOME

by

G. E. Goheen, Assistant Director
Southern Utilization Research and Development Division

It is both a pleasure and a privilege to welcome so many representatives of the cotton textile industry and related industries to this Fourth Cotton Utilization Research Conference. The reason this pleasant duty fell to me is because Dr. Fisher had to be in Washington; otherwise, he would be speaking to you. He asked me, however, to convey his regrets that he cannot be here to meet with you, and to extend his best wishes for a very fine conference.

When the Southern Laboratory held the first of these conferences in 1961, we were highly gratified by the interest shown by industry through their attendance at the meeting. It is even more gratifying to see so many here today, and especially to see so many who have been attending every year.

This interest in utilization research, not just this conference, but all kinds of utilization research, is a healthy sign for the cotton textile industry. For many years its members enjoyed the pleasant position of being able to go along year after year, producing much the same kind of products in much the same manner. The advent of rayon, however, put an end to this period of security, and the rapid increase of competition from a host of synthetics and other materials during the postwar years jolted the cotton industry out of any remaining attitudes of complacency.

For the consumer, this has been a good thing; right now he is being offered a wider choice of better products than was ever before available in history. The situation has not been so comfortable for the people engaged in the manufacture of products from cotton, but certainly cotton is not the only natural product faced with this kind of competition. Wool and silk have been confronted with the same situation about as long as cotton. Leather is the latest of the natural products to face a serious attack in a market where for centuries it has been the standard, and has enjoyed a monopoly. The first of the synthetics which claims to be better than leather came on the market a few months ago, and reports are that at least one more, and maybe still others, will make their debut soon. It's an ill wind, however, that blows no one good, as the old saw has it; we have been reliably informed that stretch cotton fabric is being used as a backing, or lining, for the first of these new synthetic leathers to reach the market. This could lead to the recovery by cotton of at least part of the market lost when manufacturers began leaving linings out of shoes, especially those for women. One thing is certain--that cotton, leather, and many other natural products will never again enjoy a monopoly as a matter of course--these commodities will have to fight to hold their position in the market.

Perhaps in a way it has been fortunate that the rivalry of other materials with cotton started as early as it did, before the great surge of technological advances that occurred right after World War II. Research on cotton started slowly, but it did start, and we are already seeing important results. Two developments alone, first wash-wear, and now stretch cotton fabrics, indicate what can be done with cotton through research on cotton utilization. Wash-wear is no longer a novelty; it is accepted as a staple in the consumer market, and is credited with retaining for cotton a domestic market amounting to 1.2 million bales a year.

All-cotton stretch fabrics have been on the market for only about 2 years, but when manufacturers report they are turning out these stretch cottons at the rate of 400,000 or 500,000 square yards a week, stretch cottons are definitely a part of the scene. Preliminary reports indicate that the volume on stretch fabrics in 1963 was roughly $600 million. By 1965, it is predicted the market will top $1 billion and reach several billion by 1970. Cotton should have a liberal share of this market, and we believe that it can get it.

Altogether, utilization research on cotton by the USDA and other organizations is believed to be responsible for the sale of approximately 2 to 2-1/2 million bales of cotton annually, or about one-fourth of the domestic consumption.

Cotton is the object of most of the research effort at the Southern Regional Research Laboratory. In 1963, approximately 60 percent of our total effort, or 165 man-years, was devoted to cotton, and 13 percent, or 37 man-years, to a closely related product, cottonseed. For the current fiscal year Congress appropriated increases for construction, alterations, and new equipment at the Southern Regional Research Laboratory and for contracts and grants. We are going ahead with plans to expand and improve our research program.

In a recent address, Dr. Byron T. Shaw, Administrator, ARS, stated some of the policies of the Department and the Agricultural Research Service, when he declared: "... the most important single need for change and improvement in agricultural research if we are to meet the challenges of the future successfully... is our current and continuing emphasis on basic research."

Here in the Southern Division we are definitely following this trend in regard to cotton. Our goal is a research program divided about half-and-half between basic and applied research, so as to provide a flow of fundamental information essential for the more sophisticated type of research that is going to be required in the future, while continuing to give attention to the development of new products and new processing methods.

Another change taking place at an accelerated rate is the expansion of our research through contracts and grants. As the fund of scientific knowledge becomes broader and deeper, it becomes increasingly clear that better research results can be obtained through closer cooperation in larger scientific communities, with several disciplines working together. This is true of the Southern Division, as well as the Department as a whole. This is being accomplished through contracts, grants, and cooperative agreements with colleges and universities, with private research organizations, industry, and the land-grant colleges and state experiment stations. Cotton is receiving much of the benefit from this program.

Research under contract or grant makes available the facilities and personnel of the best research organizations in the country in specific fields, provides highly specialized equipment and personnel when needed, and also stimulates the interest of other organizations in research problems related to Southern agricultural commodities, such as cotton.

The program for the present conference gives a good general outline of what we are now doing in cotton utilization research. The first section, to be presented this morning, is devoted to fiber properties and their relation to product quality, as affected by processing variables. It is believed that you will also be interested in the report on recent developments in wool research, from the Western Division.

This afternoon we take up some results of fundamental research, and tomorrow morning various parts of our chemical finishing research program. I might call your particular attention to the paper on permanent luster, high strength, and wash-wear properties. Improved strength in wash-wear fabrics and high luster are both properties which have aroused a great deal of interest.

Stretch cotton yarns and fabrics, produced by crosslinkage and by slack mercerization, are the subject for the final session tomorrow afternoon, after which you are invited to come to the Southern Regional Research Laboratory for a tour of the facilities, and for individual conferences on any subject which you wish to discuss.

You may have noted that we have a very special speaker for the luncheon today in the person of Dr. Nyle C. Brady, Director of Science and Education for USDA. Dr. Brady was appointed recently to this newly created post, which is intended, among other things, to bring about closer coordination between the USDA research activities, the land-grant colleges, and the state experiment stations. Dr. Brady came to the Department after a long and distinguished career in agronomy, largely spent at Cornell University, where he headed that department since 1955. He has also been very active in the American Association for the Advancement of Science. We are very fortunate to have Dr. Brady on this program.

I want to thank Otto Goedecke, who served as chairman of the conference last year, for returning as Honorary Chairman this year, and Dr. George S. Buck, Jr., of the National Cotton Council, for being so kind as to take on the duties of General Chairman this year. We also appreciate the assistance of L. R. Brumby, of the Bibb Manufacturing Company; Fred Fortess, of Celanese Fibers Company; Aubrey Goodson, Jr., Dan River Mills, Inc., in serving as chairman for the various sessions; and of J. M. Cook, J. P. Stevens Company; Larry Heffner, Agricultural Extension Service, located at North Carolina State College; and Donald Gagliardi, Gagliardi Research Corporation, for presiding over the discussions.

Gratitude is due also to members of the staff of the Southern Division who have worked long and hard in making the arrangements for this Conference, and particularly Dr. Bruno H. Wojcik, Assistant Director for Industrial Development. Dr. Wojcik succeeded to this position on the retirement of Elmo L. Patton, who had active charge of arrangements for the first three of these conferences. I am sure that many of you already have at least a professional acquaintance with Dr. Wojcik from his many years with Hooker Chemical Company and with Olin Mathieson. While he has been with the Southern Division only for a short time, he has already earned the respect of all of us and I feel sure you will all enjoy working with him.

And now, with a hearty welcome, and the hope you will find the conference most rewarding, I turn the program back to Dr. Wojcik.

OPENING REMARKS

by
Otto Goedecke
Otto Goedecke, Inc.
Hallettsville, Texas

In the passing of the Agricultural Act of 1964, Congress saw fit to include 10 million dollars for research to make cotton more competitive and to allow for greater consumption of the fiber. There is naturally a great desire to give to the producer the opportunity to reduce his costs by better efficiencies in cultivation and harvesting by improved yields and quality of the fiber. Equally important in making cotton competitive in the marketplace is the efficient utilization of the fiber for it is in the finished goods where cotton finds its challenge.

Many years ago we seemed to care very little how the cotton fiber behaved in the yarn or the cloth. There was no equal to the natural fiber and cotton was taken for granted. Too long have we been imbued with the idea that cotton is good enough. Cotton is good, but in this day of modern research it must be better to stand the onslaught of its many competitors in the textile field.

The measure of our accomplishments in cotton research comes with a comparison of production yields and production costs of 10 and 20 years ago with yields and costs of today. A like comparison in the field of utilization is equally enlightening. We need only to have a look at the program of these 2 days of presentation to find an appreciation for what has been accomplished in the past 10 and 20 years.

We are living in an age of which there is no equal in history. A child born in early 1900 would have found itself at home had it been born 200 years earlier. What is even more interesting to observe is that a child born during the American Revolution could have fitted itself with ease into the area of the discovery of the Americas and with equal ease into the area of the Roman conquerors. Within a short span of half a century we have completely changed our mode of life, wherein a child of today would be completely lost if transplanted into the early 1900's. It was in 1903 that the Wright brothers flew their airplane a hundred yards. In 1911, a bold adventurer flew from St. Louis to Chicago in one day. We had no movie theater, no radio, no television, and the horseless carriage had become of age. We had no sales tax, no income tax, and the Federal deficit was less than a billion dollars. The U.S. Army had 11 planes in 1913 costing 60 thousand dollars.

Something dramatic happened in this short span of our lives. Without the research as we know it today this development could not have happened. The very foundation of our life today depends upon research as a tool to solve the complexities that this modern world presents to us.

Here at Southern Utilization Research we find a small part of this great network of modern living through research. Yet it is tremendous what this one branch of cotton utilization research has accomplished. The easy care and stretch fabric developments, along with flameproofing, soil and mildew resistance of cloth have made a great contribution to cotton's competitiveness and will continue to measure up to cotton's greatest need, a truly utilitarian fiber. With renewed emphasis on cotton research through funds made available by congressional action and the efforts of the Cotton Producers' Institute, there lies a great future ahead for this Southern branch of Utilization Research and that it will make its contribution to this modern world of living by keeping the cotton fiber an integral part in the textile and apparel field.

Keeping cotton competitive not only in price but also in quality and utility is an everlasting challenge. The people here at Southern Utilization who have devoted their time, their life to this great effort deserve your commendation as they present once more to you the results of their work. They are the vanguard of this new age which has carried us to heights never envisioned by mankind before.

We salute the Southern Utilization Research and Development Division and its faithful workers.

FIRST SESSION: L. R. Brumby, Chairman

INTERACTION OF SPINNING VARIABLES ON YARN PROPERTIES AND SPINNING EFFICICENCY

by
L. A. Fiori and G. L. Louis
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by L. A. Fiori)

Developments in the field of spinning technology are advancing rapidly, and spinning machinery is now equipped for very high drafts and spindle speeds. The need for manufacturers to produce textile materials at reduced costs is prompting a movement towards use of high drafts and spindle speeds with both warp (medium twist) and filling (low twist) yarns. It is becoming increasingly evident that spinning variables (twist, draft, and draft distribution, spindle speeds, tensions, etc.) exert a greater influence on end breakage than fiber properties when varied within practical levels, and that the combination of spinning variables which produce maximum yarn strength may not produce the lowest end breakage. To realize maximum benefits from fiber properties, it is necessary to establish spinning processing guides from which provision is made for the best combination of spinning variables "tailored" to provide an economically and technologically balanced level of yarn strength and end breakage for a given cotton.

A limited investigation was conducted to determine the interaction of the spinning variables, twist, spindle, speed, draft, and yarn number, on yarn properties and end breakage in spinning using a 1-1/16 inch cotton of average fiber properties. The cotton was spun into three yarn numbers--[30/1 (20 tex), 35/1 (17 tex), and 40/1 (15 tex)] --using three twists--3.9, 4.15, and 4.40 twist multiplier (TM)--and three spinning drafts (20, 27, 34). The SRRL 720 Spindle Hour Spinning Test was used to obtain the complete spinning performance history over a large spindle speed range.

For all yarn numbers, end breakage increased as spindle speed increased regardless of twist or draft. End breakage increased as twist decreased and draft increased. The rate of end breakage was greater for the low than high twist and for the high than low draft.

Data relating to end breakage, twist, draft, and spindle speed were combined so that changes in end breakage can be predicted from changes in spindle speed at any given constant front roll speed. For example, for a given draft of 27 and a 172 front roll revolutions per minute, a reduction of 0.25 TM (4.4 to 4.15) resulted in over a 100 percent increase in end breakage--in spite of a corresponding reduction of spindle speed of about 800 r.p.m. caused by reducing the twist.

Increases in twist ordinarily resulted in decreases in end breakage; however, end breakage could be kept at a constant level as twist is decreased by a proportional increase in spindle speed, with the rate of increase in spindle speed being greater as the yarn number becomes smaller (coarser). For example, a reduction of 0.25 TM (4.4 to 4.15), without any corresponding change in end breakage, necessitated a reduction in spindle speed from about 12,000 to 11,000 r.p.m.; or for a 30/1 yarn and constant 4.4 TM, an increase in draft from 27 to 35 required a corresponding reduction in spindle speed from 12,750 to 12,000 r.p.m. to maintain end breakage constant. Graphically it can be shown how a constant level of end breakage may be kept by selective combinations of spindle speed, twist, and draft for any given yarn number.

Generally, spindle speed showed no consistent influence on yarn strength but it appeared that strength decreased slightly as spindle speed increased over a range, in most cases, from about 11,000 to 14,000 r.p.m.'s. Average yarn package density was inversely correlated with draft for 2 out of the 3 yarn numbers. Yarn strength, uniformity, or break elongation did not correlate with end breakage regardless of twist, draft, or yarn number.

EFFECT OF HARVESTING AND GINNING VARIABLES AND FIBER LENGTH DISTRIBUTION ON YARN PROPERTIES AND SPINNING EFFICIENCY OF A CALIFORORNIA IRRIGATED COTTON

by
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Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by G. L. Louis)

Since fiber properties are often changed by harvesting and ginning practices and conditions, it is important to know what changes in spinning organizations are necessary to offset the adverse effect caused by any changes in the fiber length distribution of cotton fibers. For this purpose, this investigation was initiated using four sets of cottons of a known variety, but differing mainly in fiber length distributions induced through varying harvesting and ginning conditions. These cottons are designated according to the harvesting and ginning treatments, e.g., high moisture seed cotton, ambient drying temperature (code H-A, 4.24 percent short fiber of 3/8 inch and less); high moisture, high drying temperature (code H-H, 3.93 percent short fiber); low moisture, ambient drying temperature (code L-A, 4.05 percent short fiber); and low moisture, high temperature (code L-H, 5.04 percent short fiber). Each of these lots consisted of about 800 pounds of lint cotton and is a composite of well blended batches of seed cotton harvested at about the same time of day on 4 consecutive days. The high moisture content (12.6 percent) lots were harvested around 5 o'clock in the morning while the low moisture content (7.4 percent) lots were harvested around 3 o'clock in the afternoon. The ambient temperature during ginning was 79°F. and the high temperature 275°F. The spinning performance of these cottons was determined by the SRRL 720 Spindle Hour Test method in spinning a 40/1 (15 tex) yarn at 3 levels of spinning draft (20, 28, 35) and 3 levels of twist multiplier (3.75, 4.00, 4.25) for a total of 36 treatment combinations. End breakage and yarn property data used in the analyses were based on common spindle speed of 11,000 r.p.m. for all combinations except two in which data from the 10,000 r.p.m. were used.

ANALYSIS OF DATA

Yarn Properties

Yarn Strength. Lot H-A had the greatest single strand yarn strength while Lot L-H had the lowest and other 2 lots fell in between through the range of twists and drafts studied. In general, the cottons ginned at ambient temperature spun into yarns of higher strength than the lots ginned at high temperature. Also, cottons with high seed cotton moisture yielded yarns of higher strength than the low moisture content cotton for all yarn twists and spinning drafts used. Yarn strength did not rank in the same order according to short fiber contents when individual cottons were compared. However, when the lots were combined according to temperature or moisture variation, the effect of short fiber content on yarn strength became evident. On the average, low short fiber lots produced yarns of higher strength than the lots with greater amounts of short fibers. Therefore, reservation should be made when using fiber length distribution as the sole criterion to predict the strength of yarns spun from a cotton. Furthermore, knowledge of harvesting and ginning history of a cotton is essential in better understanding the spinning behavior of a cotton.

Yarn Elongation. The relationship between yarn elongation at break and twist and draft is rather erratic. However, drying temperature apparently exerts a greater influence on yarn elongation than seed cotton moisture, as evidenced by the sharp decrease in yarn elongation on the low moisture lot when ginned at high drying temperature; while at ambient temperature yarn elongation of the high and low moisture lots were nearly equal.

Yarn Uniformity. All the lots reacted rather erratically with respect to change of yarn twist and draft although Lot L-H was consistently less uniform than the other lots. However, when twist and draft were averaged, yarn spun from the high moisture lots were more uniform than the yarns from low moisture lots.

End Breakage in Spinning. In general, Lot L-A had the lowest end-breakage rate while Lot H-H had the highest in the ranges of twist and draft studied. Cottons ginned at ambient temperature had less end breakage than cottons ginned at high temperature, which may be attributed to the difference in short fiber contents of the lots and/or changes in surface properties and fiber damage. Furthermore, lots having low seed cotton moisture content generally spun better than lots with high moisture content. Nevertheless, end-breakage rate of these 4 cottons did not rank in the same order as short fiber content.

Observation was also made that increasing yarn twist and decreasing draft will decrease end breakage and that the rate of decrease is much greater for a cotton having more end breakage than a cotton having relatively superior spinning qualities.

SUMMARY AND CONCLUSIONS

1. Using fiber length distribution as the sole criterion in judging yarn property and spinning performance of a cotton without also considering the harvesting and ginning condition can be very misleading.

2. Yarn strength decreased with increases in draft and drying temperature but increased with increases in yarn twist and seed cotton moisture.

3. End breakage decreased with decreases in draft and drying temperature as well as increases in twist, but was not affected by changes in seed cotton moisture.

4. A cotton with inferior spinning qualities can be spun at an acceptable level of end breakage through proper selection of spinning variables, e.g., twist and draft. Decreased spinning draft and/or increased yarn twist will increase the spinning performance of a cotton and an inferior cotton will reap greater benefit from these changes than will a relatively superior cotton.

In summation, it may be concluded that the fiber properties and spinning variables which significantly affect yarn strength are not identical to those factors influencing end breakage in spinning nor do they rank in the same order of importance. Similar observation may be made between yarn uniformity and end breakage. These findings clearly indicate that yarn strength and uniformity are not sufficient to judge the spinning potential of a cotton.

THE EFFECTS OF MECHANICAL PROCESSING ACTIONS ON COTTON FIBER PROPERTIES

by
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Southern Utilization Research and Development Division

(Presented by E. A. Honold)

Some cotton properties have been found to change in value during mechanical processing. The increment of change during any one process or machine was small but, in some instances, the effect was cumulative through a series of processes. For example, the alkali swelling centrifuge value (ACV) tends to increase progressively from bale through yarn; such an increase has been considered to measure fiber surface damage. It is important that tests of cotton properties be explored for their usefulness in detecting which processing actions cause which property value changes and how these changes may affect further processing.

This investigation was concerned with the mechanical actions from opening through carding, using three cottons which represented two varieties and two ginning practices. The stock samples of these cottons had different levels in some properties, particularly in percent short fibers and strength measurements.

During the initial phase of mill processing, i. e., from opening through Axiflo cleaner to picker apron, there were no appreciable changes measured in fiber properties. The action of the picker beater was investigated by altering the beater force and the combinations of speeds of feed roll and beater to cause different beats per inch in the cotton and by passing each lot through the picker more than 1 time. At maximum action, the beater force was 33 percent more than that normally used and the beats per inch more than 5 times that of the normal action. For 3 of the properties tested, the changes in some property values were small but the trends were toward deteriorative effects as beats per inch were increased since fiber lengths decreased while percent short fibers and AC values increased.

The spinning behaviors of the cottons were in an inverse order to some of the property measurements of the stock cotton in that the end breakages in the SRRL Accelerated Spinning Test increased as the fiber lengths and strengths decreased and as the percent short fibers increased. However, while there was a similar relationship between fiber properties and breakages during spinning when comparing normal and maximum picker actions, there was no correlation among the rankings of the increments of difference in the 3 cottons.

From this investigation, we may conclude that speeding up the processing equipment without compensating factors may produce deleterious effects even though some of the fiber properties seem to be only slightly altered.

In the carding investigation, combinations of speeds of lickerin and flats produced only insignificant changes in the properties measured but, again, differences were observed in the number of breaks during spinning.

Since the variation in speed of card components did not explain the AC value increase from lap to sliver, which was found here and in previous investigations, lint samples were obtained from the running card at 6 selected points--from lickerin before and after mote knives, from cylinder before and after carding area, from doffer, and from web.

An ACV survey of these samples together with lap and sliver samples indicated the areas in which increases occurred in this property. Intracard samples were obtained from the flats card and from both of the granular cards. In the granular metallic card, significant increases were found at 3 transfer points, while in the conventional flats card, only the transfer from lickerin to cylinder produced a significant increase. It is of interest that in neither card--granular nor flats--was any change observed in the ACTUAL CARDING AREA, as was shown by a comparison of AC values for the samples from the cylinder before and after the carding area.

With transference of fibers being thus accented as a possible source of fiber surface damage, this aspect was further explored in the laboratory by transferring cotton back and forth among the 3 rolls of the Nepotometer under different atmospheric conditions of humidity and temperature. The amount of moisture sorbed on the cotton during the action definitely influenced changes in properties. With increasing dryness, an increase in fiber damage was found--more breakage, more surface damage, and slight decrease in fiber bundle tenacity. Similar effects were observed at both 24°C. and at 60°C. but, at the latter temperature, the effects of moisture were less pronounced than at the lower temperature.

Finally, the effect on AC value was investigated after subjecting cotton lint to static pressures. This inquiry arose in connection with the crush rolls, which have been recently introduced into some mills for use on card webs. Surveys were made of series of webs before and after crush rolls, and with different forces applied to the rolls. The findings were not consistent since, in some series, slight increases occurred in ACV with increasing severity of the force applied and, in other series, the changes are inconsistent and negligible. No unusually damaged areas in the web fibers were seen under the microscope. In the laboratory experiments, pressures up to 38 tons per square inch were applied to cotton lint with an hydraulic press and the AC values of the lint increased with increasing pressure. Microscopical examination of fibers after high static pressure revealed excessive compressive damage--mashing, splitting, bruising, etc.--often deep into the secondary wall.

SUMMARY

Small but measurable changes occur in fiber properties during mechanical processing. Because of the interrelation of several actions within a machine or process, the manner in which any one action affects the textile mill product is difficult to establish. In this investigation, 2 machine actions--1 in the picker and 1 in the card--were isolated as possible sources of deteriorative changes in the cotton fibers. More attention should be given to the individual mechanical actions within the machinery in order to amass and correlate sufficient data for a better understanding of the relationships between various preparatory mechanical actions and altered fiber properties. Of prime importance is the ultimate effect on the final product; whether from the point of view of behavior in chemical processing or of quality and acceptability of the textile product.

Manuscripts are in preparation describing and discussing these experiments in more detail.

SOME RECENT DEVELOPMENTS IN WOOL RESEARCH

by
R. E. Whitfield
Western Utilization Research and Development Division
Albany, California

Chemical research on wool in progress at the Western Regional Research Laboratory includes the following: polymeric finishes, fluoropolymer finishes, stretch yarns and fabrics, factors involved in the yellowing of wool, free radical chemistry of peptide bonds, steric factors in the chemistry of proteins, and various aspects of protein structure. Recent developments in polymeric finishes will be emphasized in this talk.

Three major approaches to the chemical modification of wool by polymeric materials have been used:

(1) The use of preformed polymers

Chlorosulfonated polyethylenes
Reactive polyethylene terpolymers (Surlyn T)
Reactive polyamides
Proteins
Other polymers

(2) In situ formation of vinyl polymers

Acrylates
Acrylonitrile
Acrylamides, etc.

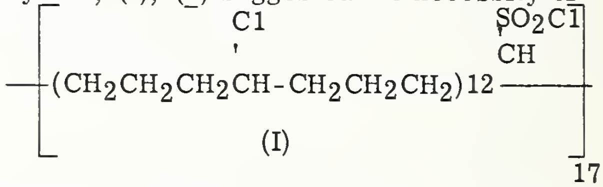
(3) Interfacial formation of polycondensation polymers

Polyamides
Polyurethanes
Polyureas
Polyesters
Polycarbonates
Copolymers

Of these, (1) and (3) will be emphasized here.

I. Preformed Polymers

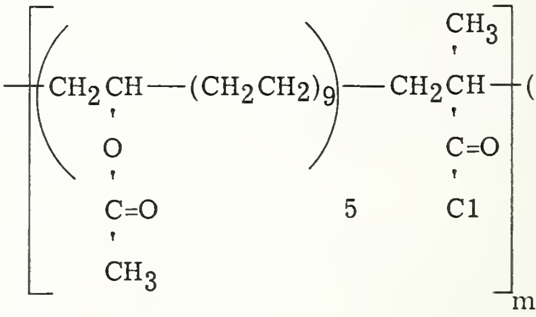
Earlier work on chlorosulfonated polyethylene, (I), (1) suggested the necessity of grafting of a preformed polymer to the wool surface to achieve permanency of the finish and to impart shrink-resistance at low levels or resin treatment. Although good shrink-resistance could be imparted to wool by grafting of (I) at 1- to 2-percent level, the cure times required (up to 4 hours) and the temperatures required (130° C.) were not of practical interest.



$$\left[-(CH_2CH_2CH_2\overset{Cl}{C}H-CH_2CH_2CH_2)_{12}\overset{SO_2Cl}{CH}-\right]_{17}$$

(I)

Recently a new group of reactive polyethylenes (II) have become available in experimental quantities. These materials, it will be noted, are quite similar to (I), but have the highly reactive -COCl groups rather than the $-SO_2Cl$ of moderate reactivity. Consequently, polymers of type (II) can be cured much more readily at lower temperatures.



$$\left[-\left(CH_2\underset{\substack{O\\C=O\\CH_3}}{CH}-(CH_2CH_2)_9\right)_5-CH_2\underset{\substack{C=O\\Cl}}{\overset{CH_3}{CH}}-\right]_m$$

(II)

These resins have been applied to woolfabric (woolens and worsted) and to wool top by a dip-pad-cure technique using chlorinated, aromatic, or paraffinic solvents. As little as 0.5 percent resin stabilizes some woolens; up to 3 percent is required for some worsteds. The modified fabrics have an acceptable hand, very good shrink-resistance, improved tear and break strengths, increased abrasion resistance, and improved resistance to hostile solutions on short exposure. Very short cure times are adequate; in some cases, simply removing the solvent by a blast of warm air near room temperature is sufficient. Application to wool top has given only moderate shrink-resistance in work so far; further study is in progress.

Earlier efforts at grafting of proteins to wool by means of formaldehyde (2, 3, 4) demonstrated that relatively cheap proteins, such as gelatin or casein, could be grafted to the wool. However, the permanency of the graft and the feasibility of the process involved in the finishing treatment left a lot to be desired. Instead of formaldehyde, we have tried several of the crosslinking agents used in cotton finishing research: dimethylol ethyltriazone, dimethylol melamine, tris (1-aziridinyl)phosphine oxide, ethylene imine, divinyl sulfone, bis(hydroxyethyl) sulfone, diacid chlorides, bischloroformates, and various reactive polymers. Our studies have been on the crosslinking of gelatin and related proteins to provide an understanding of how best to use these crosslinking agents to graft the proteins to wool.

II. Status of Research and Development on the WURLAN Process

The WURLAN process for in situ formation of polycondensation polymers on the surface of wool fibers is based on interfacial polycondensation (5, 6, 7).

(a) Evidence for graft polymer formation in the WURLAN process

A feature of the process is the formation of grafted polymer. Evidence in support of this view includes the following:

(1) The surface coating is not removed by good solvents for the coating.

(2) Blocking of the grafting sites on the wool by acetylation prior to the WURLAN treatment yields a coating which is easily extracted by good solvents; the treated wool is not shrink-resistant.

(3) When the polymer is preformed (by interfacial polymerization) and subsequently applied to the wool from solution (followed by evaporation of the solvent), the product is not shrink-resistant and the polymer is readily extracted by good solvents.

(4) By enzyme digestion of a treated wool, the protein substance can be eaten away (except for the amino acids involved in the grafting), leaving the polymeric coating. Examination of this residue from such an enzyme digestion reveals that the graft sites in the wool are the N-terminal amino groups, and the free $-NH_2$ and -CH of side chains (such as in serine and lysine).

(b) Requirements for grafting in the WURLAN process

To achieve grafting of a coating to a substrate by the WURLAN type treatment, the following conditions must be met: There must be reactive groups in the substrate; these groups should be of comparable reactivity to that of the reactive intermediates used in the WURLAN process; the groups must be accessible to the reactive intermediates of the WURLAN process; the substrate must be capable of being wetted by the treating solvents; the substrate must be free of contaminants.

(c) Factors involved in selection of a good system

The requirements for a good system for use in a WURLAN treatment are the following: fast reacting intermediates; immiscible solvents; favorable partition coefficients of the intermediates between the solvents; favorable diffusion coefficients of the intermediates; negligible competitive reactions, such as hydrolysis, reaction with CO_2, etc.; irreversible reactions are preferred.

(d) Effects of strong bases and reducing agents

Although the use of sodium carbonate was recommended earlier to take up acid formed in the interfacial reaction, recent work has shown that stronger bases are preferred and yield significantly improved results. Preferably, a base stronger than the diamine is recommended; sodium carbonate is weaker than the amines. Alkali metal hydroxides, sodium metasilicate, alkali metal salts of dithiocarbamic acids, and sodium sulfide are among the stronger bases studied. Of these, sodium metasilicate is preferred. The use of certain reducing agents in the aqueous bath of the process results in improved flat-setting and improved shrink-resistance, above and beyond that achieved in the absence of such reducing agents.

(e) Multiple-purpose finishing

By incorporation of mothproofing agents, soil repellents, water and oil repellents, pigments, etc., in either of both treating solutions of the interfacial polycondensation system, these agents get trapped in the final coating and are thereby relatively permanently fixed to the textile. Thus, while applying the WURLAN treatment, it is possible to achieve other effects simultaneously by use of the additional agents.

(f) Treatment of wool top

Extensive development work on adaptation of the interfacial system to treatment of wool top has resulted in a feasible method with certain polyamides and polyurethanes. The treated top can be processed into yarn and fabrics which possess good shrink-resistance and an adequate hand. The top treatment process is now ready for recommendation to interested mills, and for cooperative mill trials where desired.

(g) Commercial scale treatment of woven fabrics

The WURLAN process has been in commercial use for over a year. The details of equipment needed, recommended treating range, operating details, and necessary precautions for continuous long-term operations have been worked out, and will be discussed at the Technical Wool Conference to be held at the Sheraton Palace Hotel in San Francisco, sponsored by WRRL, on May 13-15. This information is available for use by interested mills.

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DISCUSSION

J. M. Cook, Moderator

Question: What sort of problems might be anticipated in respect to end breakage rate with the introduction of very high draft systems ? (300-360)?

L. A. Fiori: Today's discussion was based on drafts up to 35 on one drafting system. The very high draft systems (300-360) actually have "dual" (2) major drafting zones representing a combination roving-spinning operation. Therefore, it would appear that when the front zone draft becomes high (about 35 or over) similar effects, as discussed, would be encountered. This is based on the observation that the draft on the existing spinning frame (35) plus a draft of about 15 on the roving frame would equal a total draft of about 525 which would be more than the total draft of 300-360 on a direct spinning system. The only valid comparison is to compare the front zone draft of the direct spinning system with the total draft of the conventional spinning system.

Question: You alluded briefly to a lack of break draft data in your presentation. Is a renewed interest in break drafts arising? If so, what has caused the renewed interest?

L. A. Fiori: The trends towards higher and higher spinning drafts and recent work on break draft reported by the Clemson Pilot Plant points out a need for more information on the contribution of break drafts to end breakage in spinning so that the combination used may form a basis to obtain either maximum yarn strength or optimum spinning efficiency, or a compromise of each performance criteria.

Question: Is there any correlation of ends down with Uster "thin spots" count?

L. A. Fiori: We have never obtained a clear-cut correlation of ends down with yarn uniformity as measured by the Uster. In some specific instances there is a positive relationship, but in many cases the data are very erratic.

Question: Do the yarn strength and elongation differences observed in your grey yarns (brought about by moisture content and temperature) carry over into kier-boiled or bleached yarn?

Gain L. Louis: We did not test the single yarn beyond grey stage. However, fabric strength and elongation of the scoured and bleached fabrics made from these yarns showed that differences in yarn strength was carried over to fabric but differences in yarn elongation were not reflected in fabric elongation.

Question: Did you intend to indicate that the variations in yarn properties were associated with the percent differences in short fiber? If the short fiber had been combed out, what would you expect to get?

Gain L. Louis: We are planning to associate the variations in yarn properties with the percent differences in short fiber as much as we can. In cases where variations which cannot be attributed to short fiber content, the effect of seed cotton moisture content and drying temperature will be used to explain the variations in yarn properties. If short fibers were combed out, yarn properties probably would become very similar.

Question: Would you speculate on why HA (high moisture, ambient temperature) cotton did not spin as well as LA (low moisture, ambient temperature)?

Gain L. Louis: The slight difference in end breakage of the two lots most likely was due to the difference in trash content of the two lots. The HA was a Middling cotton while the LA was a Strict Middling cotton. Trash has a tendency to adhere to the high moisture cotton fibers.

Question: Which acid chloride gave improved handle in the Wurlan process?

R. E. Whitfield: Two combinations of diamine and diacid chloride gave improved handle.

(1) Hexamethylene diamine with dodecanedioyl chloride, i. e., C_{12} diacid chloride.

(2) Trimethylene diamine with the C_{12} diacid chloride.
Of these, (2) was particularly good.

Question: In the shrinkage tests of grey worsted flannel, were these fabrics fulled prior to treatment?

R. E. Whitfield: Yes.

Question: Who supplies the polyethylene based resins for wool treatment?

R. E. Whitfield: The resin used is available in developmental quantities (Tradename, Surlyn T) from the Polyolefins Division, Plastics Department, E. I. du Pont de Nemours & Co., Wilmington, Delaware.

Question: How do you dry the acid chloride in the Wurlan process?

R. E. Whitfield: By rapid recirculation of the bath through a small unit containing a molecular sieve. The molecular sieve takes out both HCl and H_2O; the sieve can be regenerated several times, and hence add but little to total cost of the process.

Question: Have you investigated the influence of crush rolls on the dyeing properties of the resulting cotton products?

Edith A. Honold: No.

Question: How was force of beats in pickers measured?

Edith A. Honold: The force exerted by the picker beater was not measured. But the relative tangential forces of the beater arm striking the cotton fringe can be calculated. These forces increase with increase in r.p.m. of the picker beater.

Question: What does resin-surface treatment of wool do to static electrical properties?

R. E. Whitfield: There is no apparent change in the electrical properties of the wool fiber at ordinary room conditions (temperature and moisture). However, when a treated fabric is very dry (as when emerging from a commercial dryer), it has more static than does untreated wool fabric. When the dry fabric reequilibrates with moisture from the air to attain its normal moisture content (12-15 percent), the electrical behavior is identical to that of untreated wool.

Question: Why do you use trimethylenediamine in a Wurlan type treatment?

R. E. Whitfield: Many diamines have been studied for possible utility in the Wurlan process. Trimethylene diamine (TMDA) was studied as a possible substitute for hexamethylene diamine (HMDA), which we have normally used in the Wurlan process. TMDA is commercially available, cheaper than HMDA, and with certain diacid chlorides (such as C_{12}) it gives a softer handle to the treated goods.

Question: Is an afterwash necessary to neutralize caustic in the Wurlan process?

R. E. Whitfield: Yes, an afterwash is necessary. This is done to remove unreacted reagents, the organic solvent and salts, and to neutralize the fabric to pH below 7. In addition, the process wash has a very beneficial effect on the handle of the finished treated material.

Question: Are the poly (1-perfluoroacyl) aziridines finishes merely a polymer deposited on the wool?

R. E. Whitfield: Apparently so, since they can be extracted by good solvents for this polymer. However, the polymers are fast to aqueous washing or to solvents normally employed in commercial dry cleaning.

USDA AND COTTON'S FUTURE

by
N. C. Brady
Director of Science and Education
U. S. Department of Agriculture
Washington, D. C.

The well-being of the cotton industry, from the farmer to the manufacturer, is of utmost importance to the entire National economy. In the United States, cotton is grown on about 900,000 farms and provides an income, directly or indirectly, for about 25 million Americans. Cotton, over the years, has always been the number one cash crop in this country.

The United States Department of Agriculture shares with the cotton industry a special interest and concern in the industry. A major share of the concern is in holding and improving the competitive position of cotton in the textile market.

Competition is a relatively new problem for cotton, considering the total history of mankind. Cotton is the fiber associated with civilization since its earliest beginnings. It has served man's purposes for so long that records of its first use are lost in prehistoric time.

During most of this time, cotton had no serious competition in its principal markets. Rayon made the first inroads about 35 years ago. Since that time, synthetic fibers have been appearing in increasing numbers to provide specialized qualities for different segments of the textile market.

We have all known that if cotton is to hold its own under these conditions it must be able to compete realistically with other fibers in quality and price. And the principal method of providing these requirements is through research.

The U.S. Department of Agriculture has been conducting research on some phases of cotton production since the mid-1890's. Today we are supporting a major research program affecting a wide range of the cotton industry.

Our program can be broken down into three general categories: (1) farm research; (2) consumer and industrial use research; and (3) marketing and economic research.

Let us look briefly at what USDA is doing today in these fields.

(1) Farm research includes investigations on breeding, genetics, and variety evaluation. It includes studies on the control of diseases, weeds, nematodes, and insects. We also study cotton planting, fertilizing, harvesting, handling operations and equipment, and cotton ginning.

One of the objectives in cotton breeding research is to improve the storm resistance of high yielding strains. Since harvesting has changed from a hand to a machine operation, cotton is generally left in the fields later in the season until practically all bolls are open. That means the plants are exposed to more wind and rain storms, which tend to reduce yields. Research has now shown that it is possible to develop plants with bolls, intermediate between "stormproof" and "open-bolled" types, which resist preharvest losses and yet harvest satisfactorily with spindle pickers. When these improved varieties are widely available, losses can be decreased and cost of production reduced.

The development of strains of cotton with seeds essentially free of the toxic substance gossypol has stimulated great interest in the cottonseed crushing industry. Breeders have been supplied with seeds and pollen of the glandless plant, and one or more breeders are attempting to breed a gossypol-free version of practically all the leading cotton varieties in the United States. This should help to open up much larger markets for cottonseed in the feed industry.

Research on hybrid cottons is making progress, but no recognized hybrids have yet been put on the commercial market. We do know that higher yielding hybrids can be grown. But we still have not solved the problem of cross pollination, and at this time the process is much too costly to make hybrid production possible. Our scientists are continuing further investigations to find ways of producing hybrid cotton on a practical basis.

In basic research on developing disease resistance, our scientists are making interesting progress, particularly against verticillium wilt. These critical studies have revealed unusual and previously unrecognized structural differences in the fungus that causes the disease. This finding can very well lead to the development of much needed information about the nature of resistance. Once we understand the phenomenon of plant resistance to various diseases, we will be in a better position to develop this quality more rapidly and at much less cost.

In searching for insect resistance, our scientists have shown that substances extracted from parts of the cotton plant vitally influence the behavior of the boll weevil. Some act as attractants and others as repellents. We are trying to purify and identify these substances. The objective is to find methods of breeding cotton plants that are not attractive to the boll weevil, and therefore less susceptible to damage.

In our research on the control of cotton insects, we are attempting several new approaches. These include studies on sterile male techniques, methods to prevent diapause and winter survival, and biological control agents, among many others.

As an example of the progress in this work, studies at the Boll Weevil Research Laboratory showed that specific spores are capable of infecting all larval stages as well as adult boll weevils. Infected late in the larval stage, the insect may live to transform to the adult stage but then dies shortly thereafter. Egg production was reduced drastically in diseased females. Such parasites and predators as these give promise of being highly useful in the control of insects.

Eradication of the boll weevil with a sterile male technique was demonstrated in an isolated cotton plot in one of our studies. Beginning August 1, chemically sterilized males were released at a rate calculated to provide more of the treated than normal insects in the plot. No boll weevil damage was found in the field after November 15. However, other studies with chemically sterilized males failed to affect the insect population. So we have not found all the answers yet, but the occasional successful attempts indicate a very real hope for success.

In discussing disease and insect problems of cotton products, I would be remiss if I did not call to your attention as forcibly as possible the serious problem facing all of us in connection with use of pesticides on agricultural production. The importance of these materials in producing high-quality crops is well known to all of us. On the other hand, the seriousness of residue problems to the consumer and to the public is the cause of widespread concern. It is my conviction that it will require the joint support of all of us to find a way to assure continued production of high-quality products, and at the same time, to assure the health and well being of people and livestock.

Of course, I cannot go into detail about all the new directions we are taking in the USDA farm research on cotton. These are merely a few highlights. But we do intend to continue and expand the studies in these areas.

We believe an expanded program to increase the efficiency of cotton production can pay off. We know the research in the past to increase technology and mechanization, to provide better producing cotton plants, tailored for mechanical processes, better weed and pest control--all these things have contributed to spectacular benefits in efficiency. For example, in the years 1920 through 1924--before mechanized production of cotton was fairly well started--it took an average of 296 man-hours to produce a single bale of cotton. It took about 52 man-hours to produce one bale in the years 1960 through 1962. That means, in the last 40 years research has helped to cut the equivalent of more than 30 working days off the man-hours it takes to produce a bale of cotton.

Much of this increased efficiency is from mechanization--and much of that mechanization has been in increased machine harvesting. In the years 1947 through 1949 less than 4 percent of the cotton grown in this country was machine-harvested. In other words, 96 percent was still being handpicked.

In the years from 1960 through 1962, about 60 percent was machine harvested. By 1963 over 70 percent of the cotton grown in this country was harvested by machine.

But there is still much we don't know about the best methods of preparing cotton for machine harvesting. We need to know more about the application of defoliants and desiccants--when is the best time to apply them under various conditions--when are such chemicals not necessary at all. These are some of the areas in which we will concentrate in our expanded research efforts in farm research.

(2) Recent accomplishments point the way toward almost unlimited directions in which cotton may find new and wider markets. For example, the development of molded cotton fabrics, announced last Fall, opens many new possibilities. Manufacturers should be able to speed production and cut costs of making upholstered furniture; bucket seats, and crash pads in the automobile industry; hats, shoes, and many other items in the clothing industry.

These developments should make it possible for cotton batting to better meet the serious competition of synthetic foams and foam rubber in padding applications and in mattresses. Two commercial plants have installed pilot lines for the production of the new cotton batting, and 8 additional companies are planning such installations.

Tests on automobile seat cushions at 2 major automobile manufacturers have shown that the new cotton batting at the present stage of development substantially meets their requirements for cushioning materials. Sample mattresses made with the batting are currently undergoing evaluation.

In a similar development, the commercialization of stretch cottons is advancing rapidly. Consumer demand for stretch and bulked textiles in both wearing apparel and industrial applications is increasing. Research which has imparted stretch and bulkiness to cotton products through the use of various chemical and mechanical processes is contributing significantly toward opening up new markets for cotton. Reliable estimates place the potential for stretch cottons at a figure comparable to that of wash-wear fabrics which are now utilizing over a million bales of cotton every year.

At least 9 finishers in the United States are in commercial production of cotton stretch fabrics, and several other companies are preparing samples on a pilot-plant scale. Most of the production is going into apparel, but upholstery, a slip cover material, and coated fabrics are also being produced.

In other directions, USDA utilization research has done much to aid cotton by developing improved textile processing machines; by informing the textile industry concerning ways to improve spinning performance, methods of blending cotton to obtain maximum use of the properties of available cottons, and the effect of fiber fineness, strength, length, and other physical properties on fabric properties and spinning efficiency.

A Pioneering Laboratory in utilization research is studying the structure of plant fibers. The scientists are looking first at the cotton fiber to find out how it is put together. We have known, for example, that the cotton fiber is made up basically, of crystalline regions of cellulose connected by unorganized or amorphous areas. It is through these unorganized areas that we can penetrate the cotton fiber with dyes or other chemicals used in treating cotton.

Our scientists are attacking these accessible or unorganized regions with various chemicals, trying to break down the structure of the fiber to see how it reacts. And then they attempt to put it together in a different way. They are deliberately changing the molecules of the cellulose in a progressive and organized manner.

Through such processes in this laboratory, we are building up a growing background of new knowledge about the cotton fiber. With this knowledge, the practical chemists can see better what they are working with and how to develop treatments for whatever quality they want to build into the cotton fabric.

The benefits from past research have been demonstrated in clearly definable terms. The results of utilization research conducted by all organizations--Federal, State, and industrial--are responsible for the sale of more than 2 million bales of cotton a year. That amounts to about 25 percent of the total domestic consumption.

(3) In marketing and economic research, the Department has a continuing program on the objective measurement and evaluation of quality of cotton fiber and on the evaluation and quality maintenance of cottonseed.

Part of this work includes an evaluation of various production, harvesting, and ginning practices on spinning performance and cotton quality. With the widespread appearance of mechanical harvesters and other mechanized production practices, cotton requires an increasing amount of preparation at the gin. At the same time, there has been a trend toward longer-staple, finer-fibered cottons that are difficult to clean without damage to the fiber. The general objective is to find ways to preserve the quality of the fiber as mechanized production becomes even more complex.

The research on cottonseed is aimed at finding improved methods of measuring quality. Among the results of the work is the development of a rapid and inexpensive test for grading lots of cottonseed for moisture and oil content. Our scientists have also developed a better method of determining the amount of free-fatty acids in crude cottonseed oil. This procedure can detect smaller amounts of the substance, using much smaller amounts of the oil for testing than was previously required.

I have been speaking about cotton research conducted by the U. S. Department of Agriculture. Actually, this work is on a cooperative basis. All of the research is planned and reviewed by several agricultural and industrial committees. Much of this research is conducted in cooperation with the State Agricultural Experiment Stations in the various cotton states. We frequently cooperate with the cotton industry and other private institutions. The Department plans to continue and expand this research by cooperative agreements, contracts, and grants.

In addition to these formal cooperative agreements, we have much informal exchange of information, advice, and discussions. In this way we keep in close touch with organizations interested in cotton.

As we look to the future, we can expect these communications to continue to our mutual benefit. We can afford to be optimistic about the ability of cotton to maintain its position in the textile market. While the synthetic fibers provide certain specific qualities for specific needs, no single fiber can match cotton in versatility.

Through research we can continue to streamline and mechanize cotton production, protect and improve the quality of the fiber, and find better ways to produce what consumers want at competitive prices. We must find ways to reduce the cost of producing cotton to make it more attractive to mills and consumers.

The U. S. Department of Agriculture is fully committed to increase our attention to cotton research in all phases of our program. We especially intend to increase the emphasis on basic research in order to broaden and deepen our fundamental knowledge--to extend almost beyond imagination the possibilities for cotton in the future.

SECOND SESSION: Fred Fortess, Chairman

THERMOPLASTICITY OF ACETYLATED COTTON YARNS

by
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(Presented by J. J. Creely)

The thermoplastic nature of acetylated and other chemically modified cottons has been known for some time (1, 2, 3). Because of this property, as well as other desirable features, there has recently been considerable interest in their utilization in the preparation of textile products having improved stretch qualities (4, 5, 6). As a result of this interest in thermoplastic cottons, and in reversible crosslinks for which thermoplasticity might serve as an alternative, it was recognized that more information was needed about the factors which are responsible for it, as well as techniques for its measurement. The present study was undertaken to more fully evaluate the thermoplastic behavior of partially acetylated cotton yarns as influenced by degree of substitution, temperature, and the plasticizing effects of water vapor.

The samples used in this study consisted of a 7s/1 scoured yarn of approximately 3 twist multiplier, spun from combed Deltapine cotton, and 10 acetylated samples prepared from it varying in substitution from DS = 0.77 to 2.93. They were wound, under reproducible conditions, onto cylindrical stainless steel rods, and heat-set in an oven under vacuum or in the presence of water vapor. After removal from the oven the yarns were wet out in water, to reduce the contribution to thermoplasticity measurements of hydrogen bonding, possibly formed during heating in the coiled form. The coiled loops of yarn are conditioned at 70°F. and 65 percent relative humidity and the amount of loop shortening is measured and expressed as a percentage of the original length.

The reproducibility of the technique was determined using measurements from duplicate sets of yarns heat-set under vacuum at each of three different temperatures. The standard error of the means of the duplicates was found to be between values of 0.46 to 0.51 percent.

The necessity of including the wetting out procedure in this technique is demonstrated in table 1 where the measurements were made 30 days after heat-setting. The differences listed between the values for the samples which were wetted and those which were not are found to be statistically highly significant and are attributed to a redistribution of hydrogen bonds in the amorphous regions during heat-setting. Wetting, followed by drying with 100 g. suspended from the yarn removes this effect which could confuse thermoplasiticity measurements.

The effect of degree of substitution and temperature on thermoplastic behavior of acetylated yarns was determined by heat-setting the series of test yarns previously mentioned at several temperatures ranging from 115° to 225° C. Loop shortening measurements indicated that thermoplasticity is strongly influenced by DS being slightly below DS = 1 and rising rapidly and asymptotically to maximum at about DS = 2.25. Thermoplasticity is strongly influenced by temperature. Under vacuum the effect is slight below 150° C., rises rapidly to a maximum at 175°, dips to a minimum at 180 to 185°, the glass-rubber transition point, and then rises again.

Table 1. --Effect of wetting out on loop shortening of heat-set acetylated cotton yarns[1]

DS	Percent loop shortening					
	Heated at 175°C. under vacuum			Heated at 125° C. with water vapor		
	Not wetted	Wetted	Difference	Not wetted	Wetted	Difference
0.00	2.5	1.9	0.6	7.5	2.1	5.4
.77	10.6	2.1	8.5	15.8	1.9	13.9
.94	17.1	3.7	13.4	18.5	2.4	16.1
1.11	17.5	7.6	9.9	21.7	3.3	18.4
1.33	22.4	12.9	9.5	25.2	6.5	18.7
1.52	22.0	13.8	8.2	22.5	10.0	12.5
1.59	27.5	17.9	9.6	26.5	12.7	13.8
1.95	24.2	18.3	5.9	28.7	15.8	12.9
2.20	26.8	20.5	6.3	25.4	16.7	8.7
2.66	27.5	21.1	6.4	29.2	17.5	11.7
2.93	27.9	22.7	5.2	32.1	20.4	11.7
Means	20.55	12.95	7.59	23.01	9.94	13.07
S. E.			.99			1.18
t			7.67			11.07

$t_{05} = 2.23$

$t_{01} = 3.17$

[1] Measurements made after 30 days' exposure to standard atmosphere.

The effect of water vapor on thermoplasticity was determined by heat-setting in the manner previously described, except no vacuum was applied and a sufficient amount of water was placed in the oven, which was vented to allow the excess water vapor to escape. In the presence of water vapor thermoplastic flow begins at a lower temperature and reaches a maximum of 175° C. This maximum averages 36 percent greater than that of the vacuum heated samples. At any given degree of substitution and temperature approximately the same thermoplastic effect is reached at a temperature 35° below that required for the same effect in the vacuum heated samples. The yarns heat-set in the presence of water vapor show minima at 150° and 200° C., and possibly one below 125°. The minima are interpreted as second-order transition phenomena. The percent loop shortening of the samples heat-set at 125° and 175° C., in vacuum and in the presence of water vapor are given in table 2.

Table 2.--Effect of DS, temperature, and water vapor on percent loop shortening of acetylated cotton yarns[1]

DS	125°C.		175°C.	
	Heated in vacuum	Heated with water vapor	Heated in vacuum	Heated with water vapor
0	0	0.8	0.8	1.0
.77	.8	1.7	.8	2.4
.94	.8	1.7	1.5	7.1
1.11	.8	1.7	4.9	11.3
1.33	.8	3.3	7.3	15.8
1.52	1.7	7.5	9.8	18.2
1.59	1.7	9.2	15.4	22.9
1.95	2.5	13.3	15.5	24.7
2.20	3.3	13.3	19.2	24.7
2.66	4.2	13.3	18.8	24.7
2.93	4.2	15.8	20.0	24.7

[1] Measurements made after 2 days' exposure to standard atmosphere.

It was of interest to determine whether the observed thermoplastic effect was truly reversible. For this purpose a series of samples previously heat-set at 225° C. under vacuum (percent loop shortening listed in Column A, table 3) were extended to their original length and heated again at 225° C., under vacuum. The percent loop shortening listed in Column A' indicates that although extensive crystallization had taken place in the first heat-setting that enough amorphous cellulose triacetate remained to permit essentially complete removal of the thermoplastic effect.

Table 3.--Effect of annealing upon the percent loop shortening of acetylated cotton yarns[1]

DS	A	A'	B
	Heat-set at 225°C.	Samples from A heated at 225° C. at full length	Heated at 225° C. at full length--heat-set as in A
0	2.1	0	1.7
.77	6.3	0	1.7
.94	8.6	.8	3.3
1.11	12.1	1.7	3.3
1.33	15.0	.8	5.3
1.52	16.2	.8	4.6
1.59	18.5	.8	6.7
1.95	19.3	1.7	5.7
2.20	20.4	1.7	7.3
2.66	21.0	1.7	8.5
2.93	21.5	1.7	8.3

[1] Measurements made after 30 days' exposure to the standard atmosphere.

The control exercised by the amorphous cellulose triacetate upon thermoplasticity is again demonstrated by the data in column B, table 3. Here the yarns were first heated at 225° C. under vacuum at full length (not wound on rods) and later wound and heat-set on the rods the same as for column A. The limited loop shortening observed in this case is due to crystallization and thereby reduction of amorphous acetate before heat-setting.

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SURVEY OF COTTON FIBER SWELLING IN VARIOUS LIQUIDS USING THE YARN UNTWISTING TEST METHOD

by
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(Presented by B. R. Porter)

When the weaving of cotton fabrics is complete, approximately 80 percent of subsequent processing involves some aspect of the interaction of the cotton cellulose with the components of liquid systems. For many years water has been the principal solvent employed in fabric processing. However, the continually increasing development of new chemicals designed to enhance the properties of natural fibers has evoked interest in the use of organic solvents for the reaction of many compounds with the fiber cellulose. Increased knowledge of the internal structure of cotton fibers has paralleled these developments and illustrated the importance of the type and extent of fiber swelling by the solvents used on the properties of the modified cottons.

Almost 50 years ago, Nodder reported his observations of the untwisting of single fibers of flax, ramie, jute, and hemp when suspended in water (2). These fibers all have an unidirectional spiralling of the secondary layers and untwist in a direction opposite their spiral direction when wet. Based on such observations, a method for comparative evaluation of the extent of swelling of cotton fibers in a singles yarn has been developed. Although the individual cotton fiber contains almost 50 reversals of the spiral of the secondary layers, a low-twist, singles yarn will untwist in a direction opposite to the direction of twist used in spinning when the yarn is suspended in a medium that swells the individual fibers.

Test yarns were taken from one lot of 7.5/1, 11 TPI (78.74 tex), grey yarn spun from Iquitos cotton and from portions of the same lot after scouring and after scouring and mercerizing. Each processing step was accomplished without application of any tension on the yarns. Six-inch lengths of the test yarns, one end knotted and inserted into a notch in a fixed support, were suspended in the test liquids by a 0.5 gm. weight attached to the bottom of the yarn. The number of revolutions made by the untwisting of the yarn in a 3-minute, post-immersion interval was recorded. Parallel measurements of fiber swelling by microscopical measurements of changes in width of individual fibers at random positions along their length when immersed in test liquids were made for approximately 15 percent of the liquids tested by the yarn untwisting tests. Scoured and mercerized fibers were separated from the yarns for use in the width measurements. Initial measurement was made on fibers mounted in amyl acetate. The amyl acetate was withdrawn, the test fibers flushed with the test liquid, and after 5-minute immersion time, fiber width was measured again. All swelling measurements were done at room temperature, approximately 27° C., and untwisting tests were done on yarns conditioned at 65 percent relative humidity.

When immersed in water, grey, scoured, and scoured and mercerized yarns made 28, 15, and 11 revolutions, respectively. This decrease in the extent of untwisting of the processed yarns is attributed to the torque relaxation which occurs during processing. In order to compare the relative swelling of the three yarns by the various liquids, the number of revolutions in a liquid was divided by the number of revolutions during the untwisting of the particular yarn in water. This ratio, termed "Swelling Index" (SI) is used in reporting most results. Percent increases in fiber width upon immersion in water were approximately 8.5 percent for grey and scoured fibers and 20 percent for mercerized fibers. Width measurements on fibers from each of the three yarns ranked the swelling power of the four liquids used on all three fiber types in the same order given by the Swelling Index. The liquids used on scoured and mercerized fibers for width measurements showed the same relative swelling powers by both methods.

In aqueous dilutions of lithium, sodium, and potassium hydroxides, grey yarns swelled least (range of SI from 1.02 to 1.88), scoured yarns swelled considerably more (SI., 1.40 to 3.22), and mercerized yarns by far the greatest amount (SI., 2.09 to 4.98). At equimolar concentrations, lithium hydroxide had the highest Swelling Indices, followed by sodium hydroxide and potassium hydroxide. This ranking of the swelling power of these solutions for cotton fibers has been reported for various other methods of measurement (1). The Swelling Indices of scoured yarns were determined in aqueous dilutions of the chlorides of lithium, sodium, calcium, zinc, cadmium, mercury II, aluminum and tin IV, at concentrations up to 5.0 M through cadmium chloride, to 0.1 M of mercury II, and to 1.0 M of aluminum and tin IV chlorides. The maximum Swelling Index, 1.68, was obtained in 5.0 M zinc chloride and the ranking of the other chlorides was in fair agreement with values reported for other methods of measurement of fiber swelling (1). Five-molar solutions of potassium thiocyanate gave approximately the same Swelling Index as found for zinc chloride.

When the 67 organic liquids surveyed by the untwisting technique are classified chemically and grouped by range of Swelling Index for scoured yarns, the hydrocarbons have the lowest Swelling Indices (0 to 0.1). Of the esters surveyed, only ethyl lactate, SI = 0.23, was in the medium range of Swelling Indices (0.11 to 0.99); the remainder were in the low range. Four ethers fell in the low range; the remaining 11 tested were in the medium range. The alcohols yielded similar results; however, methanol (SI = 0.56) appeared to have significantly higher swelling power than the other alcohols and the ethers. Swelling Indices of ketones, amides and amines were in the medium range with a few exceptions; only ethylene diamine showed a Swelling Index greater than 1.0 (i.e., SI = 2.9). Dimethyl sulfoxide and 90 percent formic acid gave Indices of 1.2 and 1.3, respectively. Indices for acetic acid and propionic acid were in the medium range, and for butyric acid, zero.

When the swelling of scoured cotton fibers in aqueous dilutions of the lower alcohols was measured by the yarn untwisting technique, Swelling Indices fell from the medium range of values to zero, or near zero, at alcohol concentrations of 90 to 95 percent, then increased and stayed at values from 0.95 to about 1.15 through alcohol concentrations of 5 percent. A similar dip in the Swelling Index versus alcohol concentration plots occurred for grey and mercerized yarns. However, in comparing the swelling of the three types of yarns, Swelling Indices were highest for grey yarns and least for mercerized yarns, at alcohol concentrations below 90 percent.

With the exception of the aqueous dilutions of the alcohols, all the organic liquids showed consistently lower swelling power on the grey yarns than on scoured or scoured and mercerized yarns. Swelling Indices were approximately equal for scoured and for scoured and mercerized yarns. Dimethyl sulfoxide, however, produced Indices of 0.19, 1.2, and 0.29, in grey, scoured, and scoured and mercerized yarns, respectively. The differences in swelling are not related to differences in moisture content of the yarns which were 6.2 percent for grey, 7.0 percent for scoured, and 8.1 percent for mercerized yarns.

The effects of some experimental variables were briefly investigated. Moisture contents of the yarns were varied from near zero to approximately 16 percent for grey and scoured yarns, and approximately 20 percent for mercerized yarns. The number of revolutions in water was approximately constant from near zero to 7.0 percent moisture, but a significant decrease occurred at maximum contents. In general, the revolutions made during untwisting increased with increased moisture content in tests of organic liquids. However, for some liquids the number of revolutions of yarns with maximum moisture content was less than at a 7-percent content. When the number of revolutions were counted at 15-second intervals for the first minute, and 30-second intervals thereafter, most untwisting was done in the first 30 seconds after immersion and with few observed exceptions, untwisting had ceased at the end of 3 minutes. Only in ethanolamine, did untwisting proceed at a more or less steady rate over a 3-minute interval. Variations in the length of yarn used, in the suspended weight, and in the length of yarn immersed, each caused some variation in the number of revolutions made; when the suspended weight was varied, the rate of untwisting was altered. These preliminary tests illustrate both the necessity for maintenance of constant test conditions and the possibility of variations in sensitivity of the test.

When ranking of some of the liquids according to swelling power for cotton by values obtained by the yarn untwisting test is compared to similar results from measurements by imbibition (3), benzene retention (4), and methoxyl content (5), the results by the yarn untwisting test agreed as well with results by any one of the other methods as these methods agreed with each other. It is recognized that the untwisting test may represent only the changes in radial dimensions of the fiber, but the possibility of torque relaxation by breaking of some less stable interchain bonds at only a monolayer imbibition of solvent (i.e., plasticization) should be considered. More extensive correlation with the results of methods which measure other aspects of fiber swelling are prerequisite to extensive application of the yarn untwisting test in its present state of development.

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REPORT ON THE MECHANICAL ASPECTS OF COTTON CRIMP

[Summary]

by
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A special method for measuring cotton crimp was developed in view of the statistical spread of parameters and the large number of measurements needed for their evaluation.

One of the parameters describing the crimp of textile fibers is their crimp-diameter. The mechanized method for measuring this and the other crimp parameters is as follows.

The single fibers are photographed from two perpendicular planes while stretched on an Instron tester at low loads and speeds. The photographs are taken at a number of predetermined points at the initial portion of the stress-strain curves of the fibers. The photographs are projected on a horizontal screen where a pointer of a curve-tracer is used to trace the curves; this tracing generates a voltage which is fed into a transistorized analog computer.

The computer processes this data and calculates the least-mean-square of each traced curve. The readings of two perpendicular projections then provide the crimp-diameter.

This computer system is successfully used for measurements of crimp of cotton fibers. From the stress-strain curve of each fiber the uncrimping energies and crimp ratios are obtained.

Twelve samples of cotton of different varieties with predetermined mechanical properties were tested for their crimp. The results of these tests clearly indicate that a distinction may be made between varieties according to their crimp parameters. It may be concluded from these results that the finer cottons are usually associated with lower crimp parameters.

Crimp-load relationships as well as crimp recovery parameters were investigated. For all cottons, a linear relationship was found between the reciprocal crimp-diameters and the loads, between the crimp-diameters and crimp-ratios, as well as a log-log relationship between crimp diameters and the respective stresses.

It seems that crimp is a factor influencing twistless fiber assembling, originating some bending moments appearing from interaction between one fiber and its neighbors. Therefore, when a fiber is withdrawn from a lot the contact points or areas give rise to frictional forces acting against their sliding motion. Therefore the withdrawal forces of raw lots of the same cotton varieties were measured. Some correlation between these forces and the crimp parameters is indicated, supporting the former assumption.

Alkali treatments of cotton fibers were performed and the formation of more crimped samples was observed. These were tested and the crimp diameters, as well as withdrawal forces, indicate a highest crimp region around 18-20 percent of alkali.

RATE OF DRYING OF CHEMICALLY MODIFIED COTTON FABRICS

by
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(Presented by H. J. Janssen)

The chemical engineering concept of drying is that drying proceeds in two distinct, consecutive periods: The constant rate period and the falling rate period. The constant rate period exists, and continues to exist, so long as water reaches the fabric surface in sufficient quantities to equal the rate of evaporation into the surrounding atmosphere. The constant rate depends principally upon the environmental drying conditions of temperature, relative humidity and air movement and, to a lesser extent, upon the fabric structure. Eventually, some critical moisture content is reached wherein the plane of evaporation becomes partially subsurface and the drying rate begins to fall. This introduces the falling rate period. The drying rate finally diminishes to zero at a moisture content for the fabric sample which is in equilibrium with the drying air. The falling rate period is primarily dependent upon the morphological nature of the material being dried and, to a lesser extent, upon the drying conditions. In any practical mill operation, the greater proportion of drying time and of drying costs is consumed within this falling rate period. Yet, despite this seeming importance of the falling rate period of drying, it has not received the same emphasis in scientific research as have other phases of drying of textiles. The work reported herein is an exploratory and fundamental investigation of this falling rate period, supplemented by some analysis of the constant rate period, with the view of developing procedures for making detailed study of the morphological changes occurring within the cotton fiber as it undergoes chemical modification.

Procedure

A procedure has been developed for the accurate and precise measurement of the rate of drying of chemically modified cotton fabrics. The procedure comprises a continuous recording of the change in weight, during the drying of the test fabric, with an electronic strain gage. Drying is conducted within an environmental chamber to provide exact control of conditions in the circulating atmosphere. An electronic digital computer is employed to calculate the derivatives, or drying rates, of the experimental curve for selected time increments of drying. Using these drying rates, the computer next calculates a polynomial curve-fit for the falling rate period; a linear plot for the constant rate period; the point of intersection between these two curves as the critical moisture content; and the heat and mass transfer coefficients to permit delineation of the drying mechanism. For purposes of comparing two different fabrics, the computer is also employed to divide the region between the critical and equilibrium moisture contents into 20 equal increments of moisture; to calculate the corresponding drying rates; and to convert these values into a percentage basis. The speed of drying within the fiber for any chemically modified cotton may then be readily compared with that of the untreated control at any level of percent critical moisture content.

Results and Discussion

Constant drying rates at any prescribed conditions of the surrounding atmosphere are found to vary greatly with fabric thickness. Higher rates are found at greater thicknesses. This is principally a consequence of differences in the surface area available for evaporation and partly predicted by differences in air turbulence at the air-fabric interfaces, thereby changing the heat transfer coefficients. The slope of the constant rate line will often depart from the theoretical horizontal position and show either upward or downward slopes. Downward slopes are more common, particularly with lightweight fabrics, and reflect changes in the effective surface area of any one fabric as drying proceeds.

Drying rates in the falling rate period may be either increased or decreased by chemical modification. In general, the more hydrophobic modification treatments, and surface treatments, seem to produce an overall fiber structure which increases drying rates within the falling rate period. Among such treatments are lead chromate and partial acetylation. Hydrophilic modifying treatments and application of tension before, during, or after treatment decrease these drying rates. Decrystallization, mercerization, and hydroxyethylation are good examples of these latter modifications.

The presence of resin migration is detectable from the falling rate drying curves at drying and curing temperatures in excess of 180° C. Resin migration cannot be detected at 160° C. unless resin add-on is especially heavy. For the falling rate period, it is believed that resin migration is inward toward the finer fiber interstices rather than outward toward the fiber surface.

Theories for capillary movement of water within the fiber boundaries is not commensurate with observed experimental results. The movement of water as a result of pressure gradients during fiber shrinkage also seems negligible. Primary movement of the water appears to be by vapor diffusion if the standard equations for diffusion are modified for increasing heat flow as the drying rate falls and if these heat equations, in turn, are buffered for heats of desorption of bound water.

Summary

It is believed that the methods described herein for comparing drying rates within the falling rate period can be adopted for use in detailed investigations of the effects of particular chemical treatments on fiber microstructure to provide basic information to optimize large-scale processing variables and to improve product quality.

DISCUSSION

Lawrence L. Heffner, Moderator

Question: Is the effect of DS on thermoplasticity independent of the location of the substituent? That is, is thermoplasticity of (a) cotton acetylated directly to DS = 2.25, the same as (b) cotton acetylated to DS > 2.25 and saponified back to DS = 2.25?

J. J. Creely: The effect upon thermoplasticity of partially saponifying the acetylated cotton was not determined during the course of this study. We are, therefore, unable to say whether there would be any difference in thermoplasticity depending upon whether the DS was obtained by progressive acetylation to the desired DS or by acetylating to a higher value and then saponifying back to the desired DS.

Question: What is the precision of the measurements of the Uncrimping-energy, that is, how repeatable are the results?

Miriam Shiloh: The Uncrimping-energies are read from the stress-strain diagrams with the aid of a planimeter, as an electronic integrator is not accurate enough for the low-load-elongation regions. An error of about 5 percent in the readings of these areas is expected as a result of the drawing of slopes to the linear regions of the stress-strain curves. However, all the determinations of the Uncrimping-energies are done by taking means of at least 50 fibers from each tested sample; these means are associated with spreads reaching up to 10 percent of these. Thus it is not necessary to account for the experimental errors.

Question: What type of load cell was used in the Instron to permit measuring such small forces?

Miriam Shiloh: The "A" cell of the Instron was used for this work, as no other, more sensitive, is available yet. We have modified some of the amplifiers and other circuits in the Instron tester in order to get full scales of 250 mg. This was partly described in a letter to the editor, Textile Research Journal, 1961.

THIRD SESSION: L. Aubrey Goodson, Jr., Chairman

RAPID ELEMENTAL ANALYSIS OF CHEMICALLY TREATED COTTONS

by

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(Presented by V. W. Tripp)

The modification of cotton textiles with chemical agents to impart new and useful properties is being practiced on a wide and ever-increasing scale. A necessary parallel endeavor is the development and application of analytical methods which will provide for effective research, quality control, and product evaluation. This paper calls attention to two relatively new developments in analytical instrumentation which have been successfully applied to a wide variety of modified cottons in the determination of their elemental content. These techniques are: (1) X-ray fluorescence spectroscopy and (2) atomic absorption spectroscopy. Both are rapid in comparison to the standard "wet" methods and in most cases of equal or better accuracy.

X-ray fluorescence techniques are founded on the long-known experimental observation that when atoms are excited by radiation of sufficient energy, electrons are removed from their orbital shells. When the vacancies thus created are filled by electrons from an outer shell, X-radiation characteristic of the particular element is emitted. Measurement of the wavelength and intensity of the emitted X-rays provides qualitative and quantitative information on the elemental composition of the irradiated materials. Presently available equipment permits analysis for elements of atomic number higher than sodium.

Atomic absorption spectroscopy is used exclusively for the quantitative analysis of materials for selected elements. The method depends on the absorption of radiation of a particular wavelength (ultraviolet or visible) by a given element in the ground or unexcited state. The amount of radiation absorbed is proportional to the quantity of the element present. Atomic absorption analysis is dependent on the availability of a source of the characteristic spectral line of the element being measured, and for this reason is not as broad in scope as X-ray fluorescence.

Both methods are relatively rapid, permitting quantitative analyses in approximately one-tenth the time required by comparable "wet" chemical methods. The lower limits of reliable quantitative data from X-ray fluorescence are about 0.1 percent elemental content for the lighter elements and about 0.01 percent for the heavier elements. Atomic absorption provides reproducible data in the range of 1-10 parts per million. X-ray fluorescence is independent of the chemical state of the element, while atomic absorption requires that the atom or ion of the element be free. The X-ray technique is best applied to samples in the solid state, but in atomic absorption, it is necessary to aspirate the material into a flame.

Chemically modified cottons containing more than 20 different elements have been analyzed by these techniques. X-ray fluorescence has been applied to the determination of lead, mercury, tellurium, antimony, tin, cadmium, zirconium, bromine, selenium, zinc, copper, cobalt, iron, chromium, calcium, potassium, chlorine, sulfur, phosphorus, and silicon. While the analysis is ordinarily performed on a 300 milligram disc of ground cotton (1 inch diameter), it has been found that 4 layers of fabric of printcloth constructions will give almost identical X-ray intensity. Difficulty has been experienced in materials that have a finite particle size, such as pigments. In this case, the analytical results are only relatively, and not absolutely, indicative of the elemental content. As many as 20 analyses, involving three elements, have been performed in a total of four hours of operator time.

Zinc, cadmium, mercury, copper, and sodium have been determined on modified cottons by atomic absorption. In these analyses, the textile material is extracted with dilute hydrochloric or nitric acids, and the extract, after proper dilution, sprayed into the flame of the atomic absorption spectrometer. Attempts to analyze for calcium and magnesium have met with poor sucess, apparently because a hotter flame is required for these elements than is provided by the air-natural gas burner presently in use. Atomic absorption and X-ray fluorescence data are in good agreement in those cases where results have been obtained on the same samples.

PERIODATE OXIDATION OF FORMALDEHYDE-TREATED COTTONS

[Summary]

by
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(Presented by E. R. Cousins)

Formaldehyde is an efficient, versatile, and inexpensive finishing agent for cotton, and, as such, has received considerable attention. Three methods for treating cotton with formaldehyde at room temperature are known as the Form W (i. e., wet) process, Form D (dry) process, and Form V (vapor) process. At comparable concentrations of formaldehyde in the finished fabric, these three methods yield products which vary significantly in wet and in conditioned wrinkle recoveries, in breaking strength, in degree of insolubility, and in other properties. Such differences in physical properties of formaldehyde-treated cotton fabrics have been attributed by previous investigators to the locations and types of crosslinks. The products of these processes are, therefore, of special interest for development of an understanding of the chemistry of modification of cotton with crosslinking agents. However, to date, there is little evidence for differences in the chemical nature or reactivity of such fabrics.

The reaction of periodate with carbohydrates is well known to be specific for scission of the bonds between carbon atoms containing vicinal hydroxyl groups (Malaprade reaction). The reaction has been used to estimate the accessible regions in cotton linters and fibers. The method depends upon concurrent fast and slow reactions whose rates are determined by rates of penetration of the periodate into accessible and inaccessible regions of the fibers.

The present report is a summary of work on the feasibility of using periodate to measure the accessibility and the relative reactivities of fabrics of comparable formaldehyde content prepared by three different processes.

Desized, scoured, and bleached 80 X 80 cotton printcloth was treated with formaldehyde by the Form W, Form D, and Form V processes. Samples of a control and treated fabrics containing approximately 0. 4 percent HCHO were oxidized with approximately 0. 06 M and 0. 25 M solutions of sodium metaperiodate. The course of the periodate reaction was followed by determining the periodate consumed after various time intervals.

Analysis of the results by standard statistical procedures indicated that rectilinear portions were valid in the rate curves of the reaction of both periodate solutions with the fabrics. For the 0. 25 M run, this rectilinearity probably occurs because the periodate concentrations undergo only minor changes throughout the course of the oxidation, thus making diffusion the controlling factor in determining the rate and resulting in a zero order reaction. In the alternate case (0. 06 M run), pseudo-first-order reaction treatment is necessary to achieve straight line segments. The rectilinear sections were used to gauge the accessibilities of the various samples of cotton and to measure the rates of reaction over the linear portion of the curve.

With 0. 25 M periodate, the accessible regions of treated fabrics were reduced to 85 percent (W), 77 percent (D), and 85 percent (V) of the value for the untreated cotton; with 0. 06 M periodate, corresponding accessibilities were reduced to 88 percent, 73 percent, and 75 percent of that of untreated cotton. Thus, a small reduction in accessibility is confirmed by oxidation at both levels of periodate concentration for formaldehyde-treated fabrics and a trend in reduction of accessibility is established from cotton to Form W to Form D.

The rates of the reaction between the fabrics and the periodate solutions varied with the formaldehyde treatment. Rates of oxidation with 0.25 M periodate for formaldehyde-treated cotton fabrics were significantly reduced below that of cotton; Form W, 74 percent; Form D, 39 percent; Form V, 28 percent. Corresponding reductions in rates observed with 0.06 M periodate were 75 percent, 47 percent, and 35 percent.

The rates for oxidation of cotton with 0.25 M periodate are measures of the reaction between periodate and anhydroglucose units in the crystalline or inaccessible regions, since the measured rates cover a range of oxidation from 20 to 70 percent of the cotton, while the largest accessibility found among the samples was only about 15 percent. The oxidation rates are constant over this range indicating the controlling factor to be the diffusion of the periodate into the cellulose for reaction with vicinal hydroxyl groups. It is remarkable that rates of oxidation within the crystalline regions of cotton are controlled by chemical bonds introduced into accessible regions and that the effect is very pronounced at this low level of potential crosslinking.

Several interesting concepts suggested by a close interpretation of the data will be subject of future work.

RELATION BETWEEN SURFACE PROPERTIES OF COTTON FINISHES AND WET SOILING[1]

by
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This report is a summary and review of a series of investigations of the mechanisms of wet soiling of finished cotton fabrics that were reported earlier in detail (2, 3, 4, 5, 6, 7). The object of this work was to learn the factors that control wet soiling and ease of soil removal so that the appropriate properties of cotton finishing agents could be specified. The emphasis in these investigations was on the determination of fundamental mechanisms. This report describes the principal findings and gives the overall conclusions made from these studies.

At the outset, certain judgments were necessary regarding the properties that might be involved in the adhesion of soil to fabrics. The thermodynamics of interfaces is the area of principal importance in the adhesion theory; that is, in order to form a stable, adherent interface between two materials, the surface-free energy of this interface must be low compared with other possible interfaces that can be formed by the system. For example, if the interfacial-free energy between a soil particle and a fiber is high compared with the water-fiber interfacial free energy, the soil particle is not likely to become attached to the fiber. For this reason the surface energy of the finishes is one of the properties included in this study.

Another basic factor in adhesion is the area of contact between the two materials. Since the strength of the adhesive bond depends on surface interactions in the interface, its overall strength will be proportional to the area of contact. A large area of contact between the soil particle and the fiber would be fostered if the surface of the fiber were soft so that the particle could embed. Therefore, the hardness of finishes was measured.

Suspended soil particles and textile fibers may be considered a colloidal system. In aqueous colloidal systems, coulombic repulsion between particles due to overlapping of their electrical double layers is an important factor in preventing aggregation (8, 11). A useful method of characterizing this double layer is by means of electrokinetic measurements from which the zeta potential can be calculated. The zeta potential is the potential drop from the immobile solvated layer of the solid to the bulk of the solution. Since our interest is woven fabrics, the streaming potential appeared to be the appropriate type of electrokinetic measurement.

Methods

The surface energy or surface tension of a solid interface cannot be measured directly but relative surface energies can be obtained from the relative wettability of solids by means of contact angle measurements. Since our interest was wet soiling, the energy of water-solid interfaces was studied. The techniques used are described in an earlier report (5). Hydrophobic and oleophobic finishing materials, such as silicones and fluorocarbons were of particular interest. The preparation of these and subsequently mentioned treatments are described in another paper (2).

The hardness of polymer finishes was measured by means of Durometer and Sward Hardness tests (4). The adhesion of pigment particles sedimented onto films of the polymers immersed in water was also measured (4). The changes in hardness and pigment adhesion with temperature were measured. Silicone and fluoroacrylate polymers and a series of hydrocarbon acrylate copolymers varying in hardness were included in this study of the relation of plastic character to wet soiling and soil removal.

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[1] A report of work done under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act. The contract is being supervised by the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

The electrokinetic or zeta potentials were determined by the streaming potential method on specially prepared fabrics that might be expected to vary widely in the magnitude and sign of the potential. Aminized and phosphonomethylated cotton, carboxy and amino acrylate coatings, carboxy methyl cellulose and malamine-formaldehyde treated cottons plus some of the hydrophobic and oleophobic coated fabrics were placed directly in a streaming cell and their zeta potentials determined in dilute KCl (7).

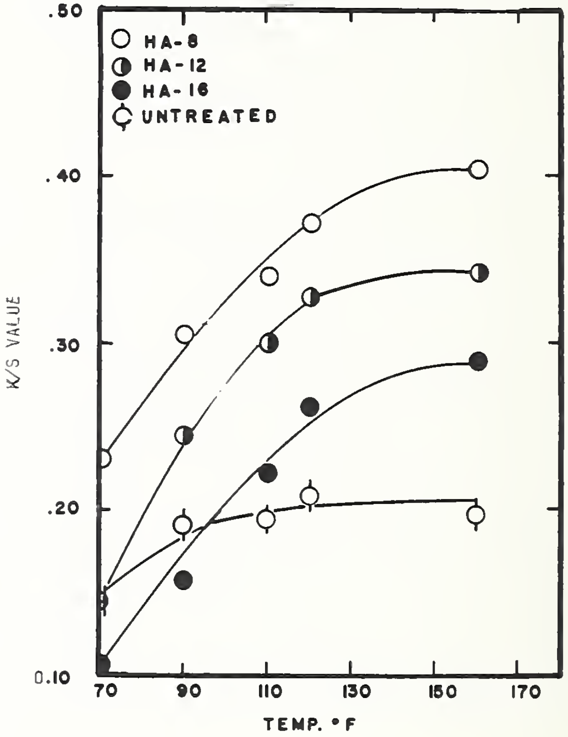
Finally, an assessment was made of the proclivity for each of these variously finished fabrics--(1) to pick up water suspended particles (2) and (2) to be cleaned in a simulated laundering procedure (3). Special attention was given to the choice of particulate soils to aid in establishing the mechanisms of soil deposition and removal, *i. e.*, iron oxide as representative of hydrophilic, inorganic soils, and carbon black and iron oxide-oleic acid mixture as representative of hydrophobic soils. Determination of the amounts of soil on fabrics was made by use of reflectance data and the Kubelka-Munk equation.

Discussion of Results

Hardness

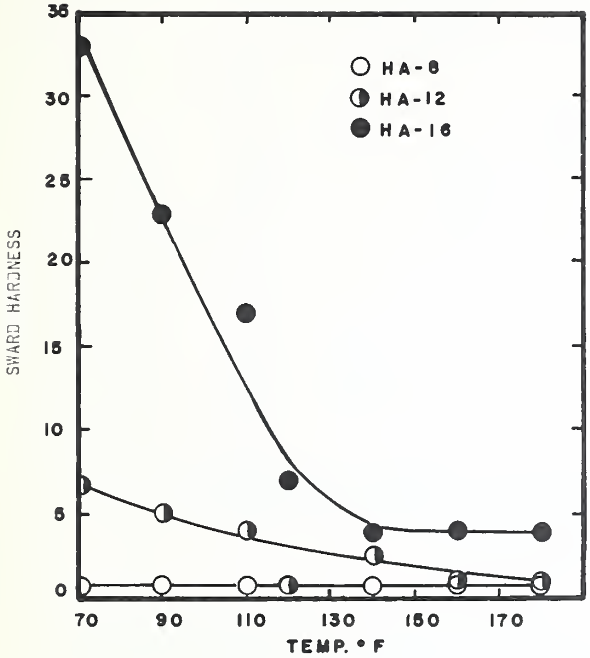
A series of finishes consisting of copolymers of methyl methacrylate and ethyl acrylate, which varied in hardness in proportion to the amount of methyl methyacrylate present, were examined for their tendency to pick up iron oxide pigment over a range of temperatures. The Sward hardness was determined over this same range. The results of soiling and hardness tests are in figures 1 and 2, respectively. Referring to the soiling data in figure 1 the polymer finished fabrics all undergo increased soiling with rising temperature but untreated cotton remains essentially constant. Comparing the hardness data in figure 2 with the soiling data in figure 1, it is clear that the softest finish is the most heavily soiled and a decrease in hardness with increasing temperature is accompanied by increased deposition of iron oxide. A Sward hardness of about 7 appears to be critical; below 7, soiling of polymer finishes becomes greater than untreated cotton.

Figure 1.--Iron oxide wet soiling as a function of temperature for cotton fabrics finished with acrylic polymers of different hardness.



In order to obtain a direct indication that the soft polymers were embedding pigment particles, a variation of the Von Buzagh adhesion test (12) was performed on films of the polymers. This consisted of sedimenting iron oxide suspensions onto the polymer films at various temperatures and determining by reflectance measurements the amount of pigment adhering after rinsing. The results for the three acrylic copolymers are shown in table 1. The amount of adhering pigment increases as the softness increases and it also increases between 70°

Figure 2.--Sward rocker hardness vs. temperature for acrylic ester copolymers.



and 120° F., in the same range where a maximum of softening occurs. Adhesion decreases again at 160° which may be due to a combination of a leveling off in softening and a decreased amount of sediment caused by more vigorous thermal motion overcoming the settling effect of gravity. These sedimentation experiments are a clear demonstration that water-suspended pigment adheres to soft polymer finishes and that the effect increases with increasing softness.

As might be expected, soil is more difficult to remove from these soft coatings than from untreated cotton. The percentage of iron oxide soil retained after laundering by untreated, hard, medium, and soft acrylic polymer-coated cotton fabrics was 26, 43, 51, and 58 percent, respectively (3).

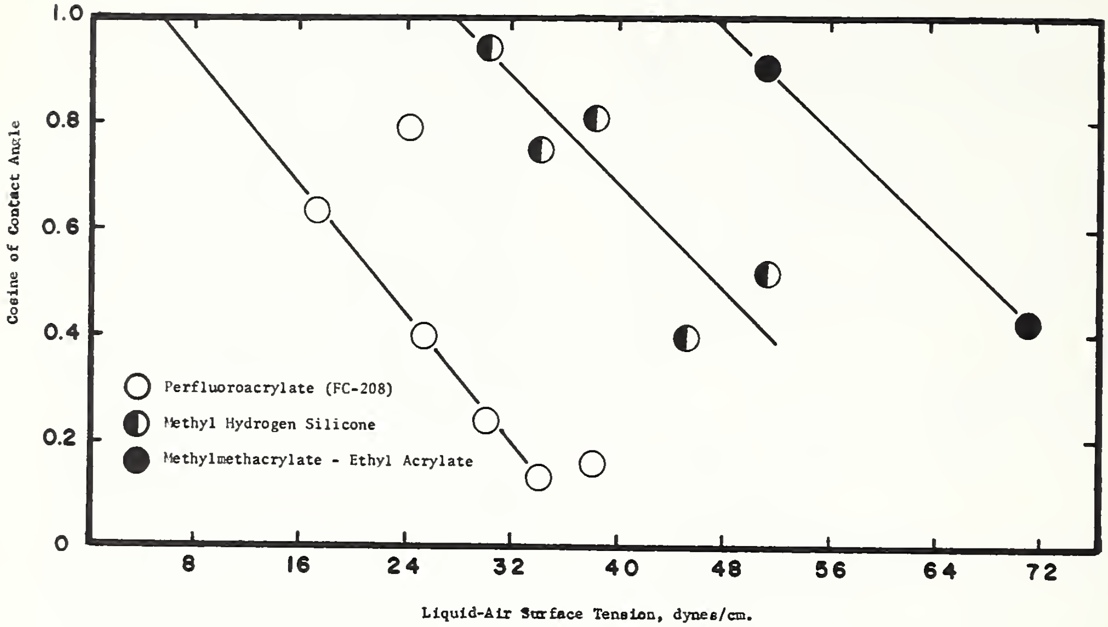
Surface Energy

The usefulness of water and oil repellent finishes for textiles is due to the inability of liquids to spread on the surface of the coated fibers and yarns which, in turn, is a consequence of the low energy surfaces formed between air and the finish. Zisman (13) has developed a method for indirectly characterizing the surface energy of solids by use of the critical surface tension for wetting. The contact angles of drops of a series of liquids varying in surface tension are measured on the solid and the data plotted as cosine contact angle vs. surface tension. The resulting line is extrapolated to zero contact angle (cosine = 1) which gives the surface tension of a liquid that will just spread on the solid. This method was used to characterize the surface energies in air of several materials used for finishing cotton. The cosine contact angle vs. surface tension data for a perfluoroacrylate, methyl hydrogen silicone and methyl methyacrylate ethyl acrylate polymer films are shown in figure 3. The critical surface tensions for wetting and surface energies of the three polymers are in the order: fluoroacrylate, silicone, hydrocarbon acrylate. Water spreads on cellulose which places cotton at the high end of the surface energy series with a critical surface tension for wetting greater than 72 dynes/cm. These results show why the fluorocarbon polymer makes an effective oil and water repellent finish and the silicone is only water repellent. The 48 dyne/cm. critical surface tension for the acrylic finish is inadequate for water repellency because of the capillary suction in the yarns which causes water to wet a fabric despite its not wetting a flat film.

Table 1--Adhesion of sedimented Fe_2O_3 pigment to polymer films at various temperatures

Polymer films	K/S value (amount of pigment adhering)		
	70° F.	120° F.	160° F.
Rhoplex HA-8 (Soft acrylic)	0.170	0.205	0.128
Rhoplex HA-12 (Medium acrylic)	.075	.123	.084
Rhoplex HA-16 (Hard acrylic)	.032	.097	.037

Figure 3.--The critical surface tension for wetting of polymer films.



Wet soiling and soil removal take place while the fabric is immersed in water; therefore it was of interest to characterize the surface energy of the finish-water interface. This was done by measuring the contact angles of drops of various organic liquids against films of some polymer finishes immersed in water (6). The cosine of the contact angle was plotted against the water-organic liquid interfacial tension as shown in figure 4. The data for the three polymers do not fall on parallel straight lines but they do show that the silicone-water interface is most readily displaced by organic liquids because the contact angles of the oil drops against this film are smallest. The contact angles against a cellulose film were very large (about 150°) for all of the organic liquids. These data indicate that the hydrophobic materials form a high energy surface with water and that hydrophilic cellulose has a low energy surface in water. In other words, the situation in water is opposite from that in air.

Insofar as surface energy considerations are concerned, the mechanism of the deposition of a soil particle from water onto a fiber can be represented by the diagram in figure 5. The pigment and fiber will each have a characteristic surface free energy or interfacial tension in water, γw-p and γw-f. When the particle becomes attached to the fiber, a pigment-fiber interface is formed at the expense of water-fiber and water-pigment interface. If this is to be a thermodynamically spontaneous process, it must be accompanied by a decrease in free energy or the sum of γw-f and γw-p must be greater than γp-f. These conditions will be most likely satisfied if γw-f and γw-p are large and γp-f is small, for example, in the case of a hydrophobic fiber and a hydrophobic pigment.

Figure 4.--The critical interfacial tension for wetting of polymer films immersed in water.

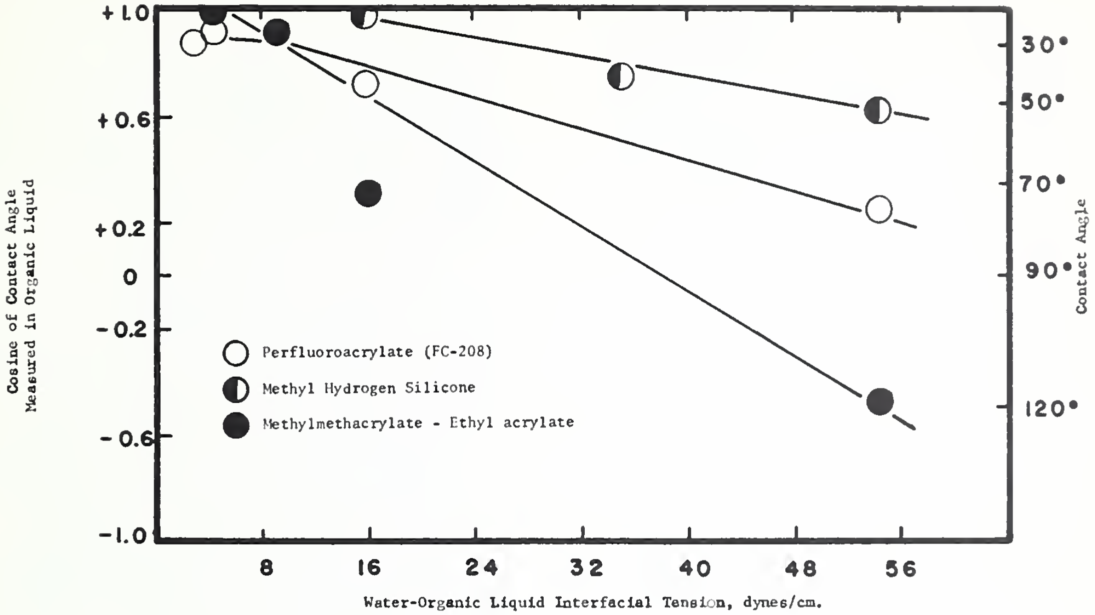
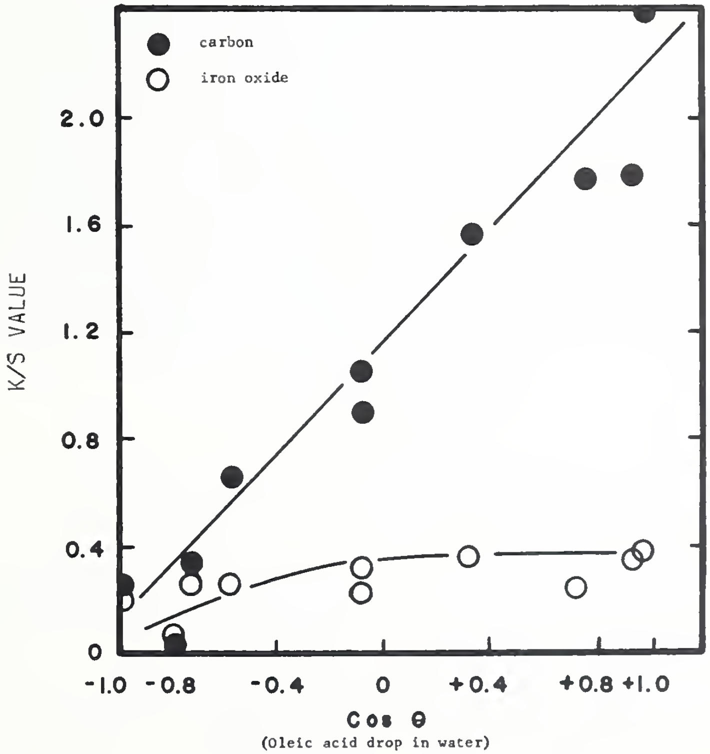
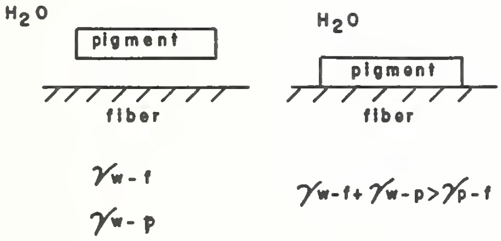


Figure 5.--Fabric soiling vs. preferential wetting by oil of fabric finishes.



A test of this hypothesis can be made by comparing the degrees of soiling of a series of cotton fabrics, with the finishes and soils differing in hydrophobicity. The cosine of the contact angle of oil against the finish immersed in water can be taken as a measure of hydrophobicity; when the cosine of the angle is large there is a strong tendency for oil to displace water. The hydrophobic soil was carbon black (1) and the hydrophilic one, ferric oxide. The degree of soiling plotted against cosine contact angle of oleic acid in water for a number of finishes is shown in figure 6. The data for carbon black show a 1:1 correlation of soiling with finish hydrophobicity, but there is very little correlation of hydrophobicity with iron oxide soiling. Some of the correlation of carbon soiling with finish hydrophobicity must be fortuitous since other factors are almost certainly operating. Nevertheless, the data in figure 6 provide strong support for the thermodynamic mechanism of soiling sketched in figure 5.

Figure 6.--Mechanism of soiling.



A separation of the hardness and hydrophobicity factors can be made by comparing soiling effects on fluoroacrylic polymer and chromic complex perfluorooctanoic acid finishes on cotton. The polymer finish is a soft, thermoplastic material which, as determined from Sward hardness tests over a range of temperatures, undergoes appreciable softening between 120° and 140° F. The perfluoro acid chromic complex is not a film forming polymer and consequently should not alter the hardness of the fibers to any appreciable extent. Soiling tests were performed with iron oxide at 120° and 160° F. and carbon at 120° F. The results are given in table 2. Referring to the data on iron oxide, the flouroacrylate is more heavily soiled than untreated cotton, and there is a large increase in iron oxide deposition at the higher temperature. The chromic complex finish picks up iron oxide soil to about the same degree as untreated cotton. There is no appreciable change at the higher temperature. The data for carbon black show that the hydrophobic, chromic complex finish is more heavily soiled than cotton and the hydrophobic, plastic finish is more heavily soiled than the chromic complex. These results in table 2 show that a hydrophilic soil, such as iron oxide, adheres to a soft, plastic finish and the effect becomes greater at a higher temperature where the polymer is softer. A hydrophobic, nonplastic finish reacts to a hydrophilic soil in the same way as untreated cotton. In the case of hydrophobic carbon soil, both fluorocarbon finishes become heavily soiled. The driving force causing deposition of carbon on the chromic complex can be ascribed to the thermodynamic surface energy mechanism described in figure 5 since the embedding effect is absent.

Table 2.--Soiling of plastic and surface reacted fluorocarbon finishes

Finish on fabric	K/S value		
	Iron oxide		Carbon black
	120°F.	160°F.	120°F.
Untreated cotton	0.21	0.18	0.38
FC208 Fluoroacrylate	.29	.44	1.78
Chrome complex Perfluorooctanoic acid	.20	.19	1.04

Another example of the interplay between the surface properties of the fabric and the soil was found in the effect of adding a small amount of oleic acid to iron oxide pigment. The surface of iron oxide is basic, which results in the formation of a chemisorbed fatty acid film. This film produces a hydrophobic surface on the mineral. When cotton fabric is agitated with a suspension of this highly hydrophobic soil, almost no deposition takes place (K/S value = 0.01). On the other hand, when the fabric is coated with a slightly hydrophobic, acrylic finish, substantial deposition of hydrophobic, oleic acid-coated iron oxide occurs (K/S = 0.30). The remarkable resistence of cotton to deposition of a strongly hydrophobic soil is understandable in view of the high contact angle of oil against cellulose immersed in water. The angle is nearly 180°, which makes it difficult to attach a drop of oil. This strong resistance of cotton immersed in water to wetting by oil is an important factor in its wet soil resistance.

Soil Removal--Relative surface energy considerations are important in the question of soil removal because the process involves the displacement of one interface and formation of two new water interfaces. Ideally, this process should take place spontaneously by preferential wetting but in practice mechanical agitation is required. An approximation of the tendency of a detergent solution to displace a hydrophobic soil can be made from the receding contact angle of an oil drop (6). The receding contact angles of paraffin oil drops on various films immersed in detergent solutions are compared with percent oily soil removal in table 3. The polymer finishes in this comparison were of approximately equal hardness. The percent oily soil removal is in direct relation to the receding contact angle.

Table 3.--Receding contact angles on polymer films in system: paraffin oil-detergent solution[1]

Polymer film	Receding contact angle	% Oily soil removal from finished fabric [2]
Cellulose (cellophane)	117[3]	96
Hydrocarbonacrylate (Rhoplex HA-12)	87	85
Perfluoroacrylate (FC-208)	66	60
Methylhydrogen silicone (ET4-0011)	32	41

[1] 0.4 percent of detergent composed of a mixture of sodium dodecylbenzene sulfonate, sodium tripolyphosphate, sodium carboxymethylcellulose, sodium silicate, and sodium sulfate.

[2] Fabric soiled with iron oxide--oleic acid mixture.

[3] At times drop was entirely displaced by detergent solution.

In the case of fabrics made with cellulose chemically modified by hydroxythylation or phosphonomethylation, soil deposition and soil removal did not have the expected relationship. These chemical modifications increased resistance to soiling (2) but they had a deleterious effect on soil removal (3). See table 4. A substantial amount of swelling and gelatinization occurred in water which upon shrinkage and collapse during drying may have trapped soil. In view of this, hydroplasticity is probably a property that should be avoided in textiles to minimize soil retention.

Table 4. --Iron oxide and carbon black soil deposition and removal from chemically modified cellulose fabrics

Fabric finish	Soil deposition K/S value		Soil removal, %	
	Fe_2O_3	Carbon	Fe_2O_3	Carbon
Untreated cotton	0.21	0.38	74	51
Hydroxyethylated cotton	.04	.37	40	34
Phosphonomethylated cotton (sodium salt)	.02	.07	62	15

It is interesting to explore the question of the relation between the tendency of a hydrophobic finish to become wet soiled and the ease of soil removal. As pointed out above, perfluorooctanoic acid chrome complex finished cotton became soiled with iron oxide to the same degree as untreated cotton. However, soil removal tests showed that more than twice as much soil remained on the hydrophobic finish after laundering (3). We may conclude from this that nonplastic hydrophobic surfaces are only subject to wet soiling by hydrophobic soils but both hydrophobic and hydrophilic soils are difficult to remove.

To conclude this section on surface energy effects in soiling and soil removal, it is clear that cotton has nearly ideal properties in this respect and that finishes which make cotton hydrophobic promote wet soiling and retard soil removal.

Zeta Potential

Theories of soiling and soil removal involving electrostatic attraction or repulsion are popular because of the ubiquity of electrokinetic phenomena and the success of the Verwey-Overbeek-Derjaguin theory in explaining colloid stability in other systems. Under the conditions encountered in practice, most soil particles (9) have a negative charge in water which would lead to the expectation that increasing the negative charge on the fabric should retard soil deposition and promote soil removal. Conversely, a positively charged fabric should attract soil and retain it tenaciously. In order to obtain a measure of the importance of electrical phenomena in soiling, fabrics were prepared containing potentially anionic or cationic groups. The electrokinetic or zeta potentials of these fabrics were measured by the streaming potential method and the results compared with soiling and soil removal data. Some of the other finished fabrics having especially strong soiling and soil retention qualities were also examined in the streaming potential investigations to learn if electrical factors were contributing to these undesirable properties.

Since the primary interest in this work was fabric finishes, it was important to be able to perform electrokinetic measurements directly on fabrics. The streaming potential technique was adopted to meet this need by using a special cell (7) and filling the space between the electrodes with a porous cylinder formed of discs of fabric. Measurements were carried out in dilute KC1 on fabric specimens previously equilibrated in this electrolyte medium as described in an earlier report (7).

Iron oxide and carbon black are known to have negative zeta potentials (9), therefore it was not necessary to measure them. Soiling tests were then made on fabrics having had the same treatment. In this way, it was assured that during the soiling tests the fabrics had the same zeta potential they had during the streaming potential studies.

The zeta potentials of the fabrics and the amounts of iron oxide deposited on them are listed in table 5. Despite the fact that the zeta potential of the fluoroacrylate finish is more than double that of untreated cotton, iron oxide deposition on it is large. Certainly factors other than the zeta potential are controlling pigment deposition. It was shown that these factors are the softness of the coating and the high energy surface formed between this hydrophobic coating and water. Similar but less extreme results were obtained with the plastic, hydrophobic silicone and acrylic ester coatings.

For the acrylic coatings containing ionizing groups, the data show a greater deposition of iron oxide on the anionic, carboxy acrylic than on the neutral ester coating. This result is also not consistent with their zeta potentials and may be due to a difference in hardness. Slightly more iron oxide has deposited on the amino acrylic than on the oppositely charged carboxy acrylic. The positively charged coating would probably have shown a larger difference from the negatively charged carboxy acrylate in pigment deposition if it had been equal to the other in softness, but the amino acrylic was slightly harder, thus tending to minimize the difference in soiling.

The foregoing experiments did not allow a clear distinction of the effect of electrical charge because of the simultaneous effect of the softness of these thermoplastic polymers. The chemically grafted aminized cotton should offer a less ambiguous test of the effect of charge on pigment deposition from water since no plastic coating was applied to the fibers. The high levels of iron oxide deposited on the three aminized cotton fabrics (table 5) can, therefore, be attributed to the attraction of negatively charged iron oxide particles to the positively charged aminized cotton fibers. Finally, the melamine-formaldehyde resin treated cotton has about the same degree of iron oxide deposition as untreated cotton, consistent with the fact that its zeta potential is approximately equal to that of untreated cotton. The amino groups in melamine did not cause a positive zeta potential, probably because they are tertiary and attached to a ring with aromatic character.

Table 5. --Zeta potentials of finished cotton fabrics and deposition of iron oxide pigment from water

Fabric finish	Fabric zeta potential (millivolts)	Iron oxide deposited (K/S value)
Untreated cotton	-19.0	0.15
Fluoroacrylate (FC-208)	-45.4	.36
Silicone (ET4-0011)	-26.3	.52
Acrylic ester (Rhoplex HA-8)	-20.4	.36
Carboxy acrylic (Rhoplex B-15)	-21.8	.46
Amino acrylic (JFB-306)	+16.8	.47
Aminized cotton (0.27% N)	+10.2	.45
Aminized cotton (0.43% N)	+9.4	.49
Aminized cotton (0.56% N)	+12.7	.38
Melamine-formaldehyde Crosslinking (Aerotex Resin M-3)	-21.5	.18

Some of the ambiguity in the mechanism of soiling of amino acrylate as opposed to carboxy acrylate coated fabrics is resolved to a certain extent by the results of the following experiment. The iron oxide soiled acrylate fabrics were examined in the streaming potential apparatus. The results of the zeta potential measurements on unsoiled and soiled fabrics are shown in table 6. The negatively charged carboxy acrylic fabric undergoes some decrease in zeta potential after iron oxide has been deposited. The positively charged amino acrylate, on the other hand, shows a much larger decrease in its positive zeta potential when iron oxide pigment is present. This relatively large decrease in the zeta potential on the positively charged fabric supports the idea that, to a large extent, the mechanism of soiling of this amino acrylate fabric is due to electrostatic attraction between the negatively charged soil and positive charged fabric.

Table 6.--Zeta potentials of soiled and unsoiled fabrics

Fabric finish	Zeta potential (millivolts)	
	Unsoiled	Soiled with iron oxide
Carboxy acrylate (Rhoplex B-15	-21.8	-17.9
Amino acrylic (JFB-306)	+16.8	+ 4.1

Of all the finishes examined, the most effective one for promoting resistance to soiling and ease of soil removal was sodium carboxy methyl cellulose (CMC). Therefore, it is especially important to clarify the mechanism by which this agent operates. Since CMC is a polyelectrolyte, it is possible that its effectiveness is due to an increased negative charge which it confers to the fabric. Zeta potential and iron oxide and carbon black soiling tests were made on CMC-treated fabric. Carbon soil was included because earlier work had shown that CMC was most effective in preventing deposition of this type of soil. The CMC-treated fabric used in the soiling tests was also subjected to the boiling and rinsing procedure used in preparation for the streaming potential measurements. This probably removed a large part of the water soluble treatment.

The zeta potential and soiling results are in table 7. Treatment of cotton with CMC results in a small increase in the zeta potential as might be expected from an anionic polyelectrolyte. CMC-treated cotton shows an increase in deposition of iron oxide and a large decrease in soiling by carbon black despite the strenuous extraction with boiling water. Since both of these soils have a negative zeta potential, the results cannot be accounted for in terms of electrical interaction. It is more likely that the effectiveness of CMC in nearly eliminating deposition of carbon onto cotton can be explained by an increase in hydrophilic character of the CMC-coated cotton fibers. The highly hydrated polyelectrolyte could provide a barrier to adsorption of the relatively hydrophobic carbon pigment.

Table 7. --Zeta potentials and pigment deposition from water for CMC treated cotton 10^{-4}N KCl, 25° C.

Fabric finish	Zeta potential millivolts	K/S value	
		Iron oxide	Carbon black
Untreated cotton	-19.0	0.17	0.18
CMC treated cotton	-22.9	.23	.04

The effect of CMC on soil removal parallels the soil deposition results, i. e., carbon removal is facilitated but iron oxide is not (3).

To conclude this section on electrostatic interaction, a comparison of the results of streaming potential and iron oxide pigment deposition measurements on cotton fabrics with various finishes shows that even relatively large changes in the degree of negative zeta potential of the fabric do not produce the expected corresponding change in deposition of negatively charged ferric oxide pigment. Substantial increases in pigment deposition accompany a complete reversal in the sign of the charge on the fabric, however. The finishes that increase the negative zeta potential and do not exhibit a corresponding decrease in deposition of negatively charged soil are probably more influenced in their wet soiling properties by their hydrophobic and plastic characteristics than by electrical ones.

Conclusions and Recommendations

The most effective treatment for cotton from the point of view of resistance to wet soiling and ease of soil removal is CMC. Its specificity for hydrophobic soils indicates the mechanism to be the transformation of the fiber surface to a more hydrophilic state rather than increased electrostatic repulsion.

Hydrophilic treatments promoting swelling of the fibers in water, such as phosphonomethylation, are undesirable because of the adverse effect on soil removal. This is probably caused by trapped soil occurring during collapse of the swollen fibers when they are dried.

The high-energy surface formed between hydrophobic and oleophobic materials and water is a major factor in causing deposition of hydrophobic soils from water onto fabrics. This is a property that must be recognized in water- and oil-repellent fabrics. Hydrophobic softening agents and hydrophobic synthetic fibers would also fall into this class.

Soft polymers definitely contribute to wet soiling and resistance to soil removal. The quantitative work in these investigations indicates that wet soiling begins to increase when the hardness of the polymer finish falls below a Durometer hardness (type 0-2) of about 60 or below a Sward hardness of 7. Thermoplasticity is also a factor, so that the hardness of polymers at laundering temperatures must be considered. These results confirm the pioneering work in this field by Mazzeno et al. (10).

Fibers and finishes having a positive zeta potential in water should be avoided because of the deposition of negatively charged particles by Coulombic attraction. On the other hand, there is no conclusive evidence that an increase in the natural negative zeta potential of cotton promotes a higher resistance to wet soiling.

Finally, cotton, being a hydrophilic fiber, has a rather large, inherent resistance to wet soiling and most of the finishing materials applied to it tend to increase wet soiling. The remarkable resistance of cotton to wet soiling by oily soil is shown by the experiments with oleic acid-iron oxide and the inability of an oil drop to attach to cotton in water.

An ideal cotton finish for minimizing wet soiling and maximizing soil removal would be a thin, nonswelling, permanent coating on the fiber with strong hydrophilic character.

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COTTON FABRICS COMBINING PERMANENT LUSTER, HIGH STRENGTH, AND WASH-WEAR PROPERTIES

[Summary]

by
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(Presented by A. L. Murphy)

Luster in cotton textiles presents many problems, among them being definition, measurement, securing adequate intensity, and improving the durability to laundering. Luster might be said to be a sensory property commonly evaluated visually. However, the visual rating of luster is suitable only for comparison and differs with different observers.

Workers in the past have found many things that contributed to increasing luster in cotton and cotton textiles. Cottons of fine fibers of long staple length and high strength are more lustrous than coarser fibers of shorter staple length and low strength. Some other contributing factors are yarn and fabric construction, kierboiling, and singeing. Mercerization under tension is one of the most effective methods of increasing luster in cotton textiles and has been used for this purpose for many years. Although high luster is obtained by tension mercerization of yarn, fabric mercerization is generally found to give inadequate luster, because of the low efficiency in mercerization of cotton yarns when they are restricted by the fabric weave. This is especially true when the fabric is made from singles yarn.

The object of the present research was to determine if greater luster and strength could be obtained in wash-wear fabrics by mercerizing the cotton in yarn form, weaving the fabric from this yarn, and crosslinking the fabric to make the luster durable. Five fabrics, including 2 broadcloths and 3 sateens, were made from pretreated yarns of Pima cotton. Yarn pretreatments compared were

(1) kierboiling followed by singeing and

(2) kierboiling, singeing, and skein mercerization of 3-percent stretch.

Broadcloth was made from 40/2 yarn pretreated by each method. Two of the sateens were made from 60/2 yarn given one or the other pretreatment. A sateen was also made from 60/2 kiered, singed, and mercerized yarn in the warp and 60/2 kierboiled and singed yarn in the filling. Finally, a sateen was made from 34/1 grey yarn in the warp and 70/2 Pima singed mercerized grey yarn in the filling, this yarn having been given continuous single-end mercerization at 2-percent stretch. Part of this fabric was scoured. All of the fabrics made from yarns mercerized under tension were highly lustrous. This was also true of the fabrics woven of mercerized yarn in one direction. The sateens were more lustrous than the broadcloth. It was concluded that it is not necessary to have mercerized yarns in both warp and filling to produce a lustrous fabric. Even the fabrics made from kierboiled yarn only had considerable luster. The broadcloth and sateen made from mercerized yarn had the same stiffness as the comparable fabric made from kierboiled yarn, as measured by the Tinius-Olsen method, and were more open as shown by measurements of air permeability and light transmission. When broadcloth and sateen made from unmercerized yarn were mercerized in fabric form under tension, they had less luster than the fabrics made from mercerized yarn, and they were much stiffer even though more open.

Samples of all of the fabrics were dyed with Indanthrene Blue, a vat dye. A part of the dyed and undyed fabrics were treated with 7-percent dimethylol ethylene urea (DMEU), dried at 88°C, friction calendered, and cured at 150°C. The ratio of specular to diffuse reflectance was measured on

(1) the untreated, dyed samples,

(2) the undyed, resin-treated samples, and

(3) the calendered, resin-treated, dyed, and undyed fabrics.

There was a good correlation between visual observation by several observers and the reflectance measurements. The reflectance values for the various undyed sateens made from mercerized yarn were fairly similar. An exception was the scoured sateen made from grey yarn in the warp and continuously mercerized grey yarn in the filling, which gave a higher value. The reflectance values of the sateens were higher than those of the broadcloth. There was little change in reflectance after resin treatment. There was a considerable increase in reflectance after calendering, with the broadcloth becoming nearly equal to most of the sateens. The highest ratios of specular to diffuse reflectance were given by the calendered scoured sateens.

Dyeing the untreated fabrics caused a large increase in ratio of specular to diffuse reflectance. With the sateens, the values were even higher than for calendered undyed fabrics. After calendering, the reflectance values of dyed fabrics were increased by a factor of two or greater. Values for the sateen woven from grey yarn in the warp and mercerized yarn in the filling were much higher than for the other fabrics. This was true both before and after this sateen had been scoured.

Tearing and breaking strength tests have been made on broadcloth woven from 40/2 yarn kiered, singed, and mercerized to 3-percent stretch, and also on the broadcloth made from 40/2 kierboiled yarn. It was found that the fabric of mercerized yarn had considerably increased breaking and tearing strength. After treatment with 7-percent DMEU, the fabric made from kierboiled yarn had a warp breaking strength retention of 55 percent while the fabric made from mercerized yarn had a warp breaking strength 77 percent as great as the noncrosslinked fabric of kierboiled yarn. Although the kierboiled fabric had some loss in tearing strength retention, the mercerized fabric surprisingly showed a large tearing strength increase after treatment with DMEU. The mercerized fabric also showed a slight improvement over the kierboiled fabric in dry and wet crease recovery.

By visual observation, there was a decrease in luster, after 20 standard launderings, of the fabrics made from mercerized yarns, although considerable luster was still retained. In the case of lustrous fabrics treated with DMEU however, there seemed to be only a very slight change in luster after 20 or even after 35 launderings. There was some decrease in gloss of the calendered DMEU-treated fabrics. The DMEU-treated fabrics had wash-wear ratings of 5 both before and after 20 standard launderings.

In summary, it may be concluded that the high luster and strength obtained in 2-ply yarn by mercerizing under high tension was carried over into fabric that had been woven from this yarn. Moreover, it was not necessary to mercerize both warp and filling yarns to produce lustrous fabrics. The luster obtained by use of mercerized yarns was rendered highly durable by treating the fabric with DMEU. The resulting fabric had a higher tearing strength and nearly as high a breaking strength as uncrosslinked fabric of kierboiled yarn, while at the same time showing extremely high wash-wear ratings. The tension-mercerization of plied yarn, weaving of fabric from this yarn, and resin treatment of the fabric have given cotton fabrics combining permanent luster, high strength, and excellent wash-wear properties.

RECENT ADVANCES IN THE ACID COLLOID FINISH

by
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(Presented by G. L. Drake)

Considerable research has been done at the Southern Utilization Research and Development Division on the formic acid colloid of methylolmelamine to produce a rot-, weather-, and wrinkle-resistant finish for cotton. The original work published by the Southern Division in 1959 reported the use of a freshly prepared colloid rather than an aged colloid to impart excellent rot- and good weather-resistance to cotton. The solution used to treat the cotton contained formic acid and trimethylolmelamine in a one to five molar ratio. It was shown that the solution must be used within 1 hour of preparation to obtain excellent resistance to rotting. Further work showed that the amount of resin add-on influenced the degree of resistance to microbiological deterioration and that the colloid finish was not affected by a severe water leaching test.

Investigations were conducted with acids other than formic acid in an attempt to form stable colloids and impart to cotton the degree of rot resistance obtained with the formic acid colloid. None of the other acids used were as satisfactory as formic acid. Fabrics treated with the formic acid colloid of methylolmelamine retain 100 percent of their treated breaking strength after 21 weeks in the soil burial beds, while untreated cotton completely disintegrates after 5 to 7 days in the soil burial beds. These treated fabrics have an initial strength loss of approximately 40 percent. The present work was undertaken in an attempt to decrease the strength losses due to the treatment. Several methods were used to increase the strength of the fabric treated with the acid colloid, for example, varying the curing temperatures, using a softener in the treating bath, and partially hydrolyzing the finish after it had been cured on the fabric.

Cotton was treated with the formic acid colloid of methylolmelamine and cured, without previous drying, at various temperatures ranging from 80° to 140° C. The samples were buried in the soil burial beds for a period of 15 weeks. Samples of fabric were also treated in the same manner and weather exposure studies were carried out for a period of 18 months. Samples cured at the highest temperature had the best rot resistance. For the weather exposure tests, samples cured at 100° C. had about the same resistance to outdoor exposure as those cured at 140° C. Formaldehyde analyses were obtained on the fabrics after various periods of weather exposure. After 4 months' weathering, the samples lost 50 to 65 percent of their original formaldehyde content, while the breaking strengths of the samples were higher than the original treated strength.

In an attempt to improve the tearing strength of acid colloid fabric, a softener was incorporated in the treating solution and the samples cured at various temperatures. The sample cured at the lower temperature showed a greater increase in the tearing strength than the sample cured at the higher temperature. The fabrics, both with and without softener, were buried in the soil burial beds for a period of 15 weeks. After the longer periods of soil burial, the samples treated with the softener had higher breaking strengths than those treated without softener.

Fabric treated with the formic acid colloid of methylolmelamine was partially hydrolyzed using an acid solution in an attempt to increase the strength of the fabric. The treated fabric was hydrolyzed at various temperatures for periods of time up to 9 minutes. The tearing strength of the fabric was significantly increased. To see what effect the hydrolysis had on the rot resistance of the treatment, samples--before and after hydrolysis--were buried in the soil burial beds for a period of 6 weeks. Partial hydrolysis of the finish decreased the rot resistance imparted to cotton by the acid colloid finish.

RESEARCH PROGRESS ON CROSSLINKED COTTON STRENGTH YARNS AND FABRICS

by
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(Presented by J. J. Brown)

This is a progress report on research conducted to develop stretch-type cotton yarns and fabrics using thermosetting resins combined with mechanical treatments. Various segments of the textile industry have shown considerable interest in cotton stretch yarns of this type. Therefore, research has been continued in an effort to improve processing techniques and obtain additional information that will enable the production of optimum stretch-type cotton yarns and fabrics for particular end uses.

Two processing techniques were used for imparting stretch properties to cotton yarns utilizing crosslinking agents. These are: (a) the back-twist method, and (b) the false-twist method. In the back-twist method, highly twisted plied yarns were treated with the resin solution, dried and heat-cured in this highly twisted state and then untwisted past neutral ply twist. In the other method, yarns were processed in one continuous operation on a modified false-twist machine.

Most crosslinking resins used for wash-and-wear treatments to cotton fabrics were found suitable for making cotton stretch yarns. A resin add-on of from 2 percent to 4 percent usually produced yarns with good stretch and recovery properties. Best results were obtained by scouring the yarns prior to resin treatment to improve resin penetration. Bleaching or dyeing should also be done before treating the yarns with resin.

Experimental fabrics, some having over 100 percent elongation-at-break, were woven from the textured cotton yarns. The all-cotton stretch fabrics had stretch properties similar to those woven from man-made fibers. Constructions with less than the normal number of ends and picks were used in order to allow the textured yarns room to relax and contract during the desizing and washing operations. As with stretch fabrics woven from manmade fibers, tension in the direction of stretch has to be carefully controlled during the finishing procedures to insure uniform results.

Fabric stretch was adequately controlled by adjusting the fabric structure. Filling-stretch fabrics were woven with 40 and 70 ends per inch in the loom state and the picks per inch were increased from 30 to 45. Stretch varied from about 20 percent to almost 90 percent. A change in either ends or picks per inch was equally effective in controlling the fillingwise stretch of the fabrics. There was almost a linear increase in unrecovered stretch as the stretch was increased. Fabrics with 20 to 30 percent stretch had 5 to 7 percent unrecovered stretch. Since most apparel fabrics seldom require more than 30 percent stretch, the recovery of these types of fabrics should be adequate for most uses. After-treating the fabrics with resin improved their recovery properties, wrinkle resistance and dimensional stability.

The durability of the resins used to produce cotton stretch yarns to home and commercial laundering procedures is of utmost importance, since fabric stretch is almost entirely dependent on the resin bonds. Loss of the resin would, of course, alter the size and fit of a garment made from stretch fabrics of this type and ultimately result in a sleazy, useless piece of material. Most of the resins used for producing cotton stretch yarns have exhibited good durability to repeated laundering in automatic home laundry equipment.

Conditions used in commercial laundries, however, are generally far more severe than home-type laundry procedures. These include higher temperatures, a lower ratio of water to cloth, and an acid sour treatment. Experimental fabrics woven from cotton stretch yarns produced using DMEU and a triazine-type resin at four different levels of solution concentrations (3, 6, 9, and 12 percent) were evaluated after repeated commercial-type launderings. The standard procedure for white goods not requiring ironing was used. The fabrics were laundered in individual bags and tumble dried after each wash cycle. The pH of the washing solutions was maintained between 11.8 for the soaping operation to approximately 6.0 for the acid sour. This acid sour may be somewhat milder than that used at many commercial laundries.

The fabrics were in an acceptable condition after 60 commercial launderings. Percentagewise, those woven from yarns treated with low solution concentrations of resin showed the greatest decreases in stretch. The breaking strengths of the fabrics remained about the same even though some of the resin was removed by laundering.

Future work on this project will be directed toward the development of optimum woven fabric structures for cotton stretch yarns. Contract research to evaluate stretch-type cotton yarns in knitwear is also under way. This will include the development of knitting techniques and the production of end-use apparel items that will be service performance evaluated.

Although the production of stretch-type cotton yarns and fabrics by these methods is still in the experimental stages, they have created considerable interest in the textile industry. The addition of stretch to cotton's many other desirable properties should improve the competitive position of cotton in this relatively new and rapidly expanding market.

RECENT ADVANCES IN THE PRODUCTION OF ALL-COTTON STRETCH TEXTILES BY SLACK MERCERIZATION

by
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(Presented by W. G. Sloan)

All-cotton slack mercerized fabrics having stretch in the filling direction are being produced by a number of large companies in the United States, Canada, and Europe. One company in the United States is known to be producing two-way stretch fabrics. Most of the fabrics are going into apparel and sportswear. For most of these uses 15 percent stretch with good recovery is considered adequate. Many of the fabrics being produced commercially are standard constructions while others are of modified design to improve stretch and recovery properties after slack mercerization. Two-way stretch fabrics are preferred for furniture covers, for use as the backing for plastic coated fabrics, in molded products, and in certain apparel uses.

This paper discusses briefly some of the effects of fabric structure on stretch properties, compares methods of measuring stretch and recovery, and compares properties of 1-way and 2-way stretch fabrics. Several other phases of our research on stretch have also been published (1, 2, 3, 4, 5, 6, 7, 8, 9).

Materials and Methods

Fabric Design

Experimental fabrics were woven in a plain and twill weave to determine the effect of filling yarn size and twist on stretch and recovery properties. All fabrics contained approximately the same number of 20/1 warp ends per inch but were woven in each weave with 10/1 and 40/1 filling yarns of low and high twist. The fabrics had the same warp and filling cover factors which resulted in comparisons of fabrics containing large and small filling yarns on the basis of yarn diameter rather than on a weight basis.

Mercerization Procedures

A roll containing each of the 8 grey fabrics was slack mercerized in the pilot plant for 1-way stretch in the filling direction. The warp yarns were not allowed to shrink during processing. Mercerization was in 25 percent caustic soda solution at 80° F., and the fabrics were in contact with the caustic solution for less than 30 seconds before being squeezed prior to washing. Washing and souring was done on a dye jig and drying was over cans. The fabrics were then resin-treated in a 6-percent solution of dimethylol ethyl carbamate (DMEC) using conventional procedures. Resin add-on was between 3 and 4 percent.

Samples of each fabric were also processed for 2-way stretch in 25 percent caustic soda at 80° F. for approximately 10 minutes. Mercerization, washing, and souring was done in the bottom of a dye winch so that the fabrics were subjected to no tensions during treatment. The fabrics were then centrifuged, smoothed to remove wrinkles, and dried on pin frames. This was followed by resin treatment in 6-percent DMEC, drying and curing on the pin frames, afterwashing, and drying on the frames. Resin add-ons were between 2.6 and 3.0 percent.

Tests for Stretch and Recovery

Stretch and recovery properties of the slack-mercerized and resin-treated fabrics were measured by means of a cyclic load test, a static load test, and a static extension test. The cyclic load test measures growth after repeated stretching to the same extension. In this test the samples are cycled 10 times to 15 percent elongation and growth is measured 5 minutes after the tenth cycle. The static load test determines the percent stretch and subsequent growth under a constant load rather than at a constant extension. The percent stretch is determined after 10 minutes under a load of 4 lb/2-inch width of sample; growth is also determined 5 minutes after the load is removed. The static extension test measures percent growth after the sample is extended for 2 hours at a predetermined extension equal to 85 percent of its extension after 4 cyclic loadings of 4 lb/ 2-inch width of sample.

Conclusions

The fabrics mercerized for 1-way stretch had more filling stretch and usually had better recovery than the fabrics mercerized for 2-way stretch. The 2-way stretch fabrics usually had more stretch in the direction in which the smaller yarns were running. That is, if the filling yarns were smaller than the warp yarns they had more filling stretch than warp stretch and vice versa. Filling stretch of 1-way stretch fabrics was generally increased by using small filling yarns and by using high twist filling, but there was also a corresponding increase in growth. In some fabrics as much as 30 percent easy stretch in the filling was obtained but growth was around 6 percent, which is higher than desired. The 2-way stretch fabrics had higher filling breaking strengths than the 1-way stretch fabrics. The 1-way stretch fabrics had slightly higher crease recovery angles as measured by the Monsanto method but this was probably due to slightly greater resin add-ons.

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DISCUSSION

D. Donald Gagliardi, Moderator

Question: How much time per sample required for X-ray fluorescence? For atomic absorbance? Relative amount for the two?

V. W. Tripp: For most elements, X-ray fluorescence will require about one-tenth of the time ordinarily needed for "wet methods."

Atomic absorption will require about one-fifth as much time as wet analysis, depending on the difficulty in getting the material into solution.

Question: Do you have any X-ray technique for determining halogens in cotton fabric, when the halogen content exceeds 5 percent?

V. W. Tripp: In general, the standard technique, using a 300 mg. sample disc, helium path, EDDT crystal and Cl Kα line appears to give good results up to 10 percent chlorine in cotton mixtures. There may be some self-absorption at the higher Cl contents, but the working curve will correct for this source of error. At even higher Cl contents, it may be necessary to "dilute" the ground cotton with untreated cotton and make the appropriate correction. We have not had experience with high Br or I content, and F is not susceptible to X-ray fluorescence measurements with standard equipment.

Question: All of your data was obtained by measuring the loss of $NaIO_4$ in the solution. Would the direct analysis of -CHO groups formed in the cotton have given the same accessibility values?

E. R. Cousins: It has been well established for the reaction of periodate with glycols, glycerine, etc. that there is a one to one molar relationship between the periodate used and each pair of vicinal hydroxyl groups oxidized in the substrate molecule. If an accurate and convenient method for determining the -CHO groups in cotton were available, then, theoretically, the same accessibility values would be found with either method. However, we made no attempt to determine the -CHO groups formed in the cotton.

Question: Have you tried to correlate accessibility as measured by the periodate method with other methods, such as dyeability, etc. ?

E. R. Cousins: In general, there is not a good correlation among accessibilities determined by different methods. The literature indicates that the measured accessibility will depend to a great extent on the method used, history of sample, etc. In our particular study we were interested primarily in the relative accessibilities and reactivities of the various samples and, for this, the periodate reaction was adequate.

Question: What is the effect of emulsifiable polyethylene and polyethylene polymers on soiling?

H. Peper, Jr.: Emulsifiable polyethylene finish on cotton fabric promotes the deposition of soil from water and hinders its removal. Since polyethylene is a hydrophobic polymer, this is what would be expected from the general findings of our research on the mechanism of wet soiling.

Question: Why did you choose cotton as your fabric substrate? Did you investigate this effect on synthetics? Would you expect the same effects?

H. Peper, Jr.: We chose cotton as our fabric substrate because it is frequently laundered and consequently it was important to learn the effects of finishes on launderability. We did not investigate synthetics, but we believe on the basis of our findings that if the synthetic fiber has a hydrophobic surface, there would be a strong tendency toward soil redeposition during laundering and in addition soil would be difficult to remove during laundering.

Question: Do you feel that an entrapment of soil in convolutions is an important factor in cotton soiling?

H. Peper, Jr.: Entrapment of soil in convolutions may be an important factor in dry soiling of cottons. However, our studies on wet soiling show that physical-chemical factors such as interfacial energy are of overwhelming importance in the water environment.

Question: Were your contact angle measurements made on fabrics? If so, how did you adjust for influence of capillaries and fabric roughness to obtain reproducible valid measurements?

H. Peper, Jr.: The contact angle measurements on cotton finishing agents were made on flat films of the polymers deposited on smooth glass surfaces. This eliminated the influences of capillaries and fabric roughness.

Question: What was the composition of the oily soil in your washing removal work? How does this correlate with soiling in commercial laundering of hydrophobic fibers?

H. Peper, Jr.: The oily soil was a mixture of oleic acid and iron oxide pigment. Since fatty acids are an important constituent in natural soil, we feel that this was a realistic choice.

Question: We assume your control was untreated cotton. Have you evaluated cotton samples finished with wash-wear resin with no repellent finish in your iron oxide and carbon black studies?

H. Peper, Jr.: We found that wash-wear treated cotton fabric soiled similarly to untreated cotton.

Question: What preparation of the cotton surface did you use for the wetting test?

H. Peper, Jr.: The cotton fabric used had been scoured and bleached.

Question: Were particle size and electrical charges of iron oxide and carbon black the same in these experiments?

H. Peper, Jr.: The carbon black particles were smaller than the iron oxide. They had approximately the same negative charge.

Question: Would you care to speculate on the mechanism by which carboxymethyl cellulose improves the antisoiling properties?

H. Peper, Jr.: A detailed investigation of this question demonstrated that the mechanism by which carboxymethyl cellulose improves antisoiling properties is by increasing the hydrophilic characteristic of cotton.

Question: In your report given at the TRI meeting in New York, you discussed the pretreatment of cotton yarns and weaving these pretreated yarns into fabric. It was stated that this process was more effective than the pretreatment of fabric, in preventing strength losses caused by wash-wear treatments. By "pretreatment," do you mean the wash-and-wear treatment of the yarns?

A. L. Murphy: No, the term "pretreatment" was meant to include slack mercerization and partial restretching which were applied to the yarns. These yarns were then woven into fabric and the fabric was subsequently given wash-and-wear treatments. This process was found to be more effective in preventing strength losses than if the mercerization and partial restretching were carried out on fabric, followed by wash-and-wear treatment. "Pretreatment" refers to what is done prior to wash-and-wear treatments.

Question: Can your mercerizing be carried out in normal warp sheet mercerizing processes? What stretch should be used?

A. L. Murphy: I do not know how much stretch you can get in warp mercerization, but you would need 2 to 3 percent stretch to get high luster.

Question: Why do softeners prevent loss of tearing strength? Does it prevent loss of formaldehyde?

G. L. Drake: In general softeners improve the Elmendorf tearing strength of fabrics due to a lubricating effect. Softeners of the type used to not seem to have much effect on the prevention of loss of formaldehyde.

Question: Would you kindly repeat the reason used for hydrolysis of the acid-colloid finish?

G. L. Drake: Hydrolysis studies were made in order to improve the retained tearing strength of the finished fabric.

Question: Why does hydrolysis increase the tearing strength?

G. L. Drake: It is general knowledge that tearing strength is reduced when fabric is crosslinked. Samples which have been crosslinked and then hydrolyzed have regained essentially all of their original tearing strength. These studies were based on the hope that partial hydrolysis would increase the strength and allow the fabric to still retain essentially all of the rot and weather resistant properties.

Question: Did you measure the loss of added weight or nitrogen content in the acid hydrolysis?

G. L. Drake: Loss in weight was not measured but the nitrogen content before and after hydrolysis was measured. The original nitrogen content was 4.52 percent and after hydrolysis for 3 minutes at 71° C. the nitrogen content was 4.33 percent--a retention of 96 percent.

Question: Does the acid colloid finish give protection against surface growth of mildews?

G. L. Drake: Samples of fabric that were exposed to 18 months' outdoor weathering in New Orleans did not show any noticeable mildew growth; however, there was considerable algae growth.

Question: Was fabric strength by strip or grab method? If the strip method were used, were equal threads rather than equal width used?

G. L. Drake: The strip method was used in obtaining the breaking strength. Samples of equal thread counts were broken rather than equal width.

Question: Why don't you use--as additional controls--the Arigal and Sta-Tuff treatments to see if you have accomplished anything relative to the performance of these commercially available processes?

G. L. Drake: In the Arigal or Sta-Tuff finished fabrics processed by steaming or ageing there is apparently very little reaction with or crosslinking of the cellulose; therefore, there would be very little to gain from hydrolysis studies.

Question: We have found that the Arigal process has little or no effect on the lightfastness of colors, whereas the continuous Sta-Tuff process has a definite depression of lightfastness: (1) Where does this melamine formic acid colloid stand in this respect; (2) what does it do to stiffness; and (3) does it offer any advantage over Sta-Tuff?

G. L. Drake: We have not evaluated the effect the acid colloid finish has on the lightfastness of colors but feel that it might be similar to the results obtained using the conventional methylolmelamine process. The acid colloid finish does firm up the hand of the fabric some. We feel that the acid colloid offers advantages over both the Arigal and Sta-Tuff finishes. It appears that the Sta-Tuff like the Arigal finish requires ageing for best results, thus making it inconvenient for processing on conventional equipment.

Question: In giving stretch fabric a light resin treatment, how much are tearing and breaking strength reduced?

J. J. Brown: Stretched cotton yarn with resin "add-ons" ranging from 1-1/2 to 5 percent showed strength decreased from about 18 percent to 50 percent compared to untreated yarns. Stretch yarns woven into fabric and then given an aftertreatment of resin to obtain wrinkle-resistance and dimensional stability showed from 5 to 30 percent less strength than before treating.

Question: What instrument is used to measure the percent of stretch of all-cotton stretch? Also, when measuring, if another type fabric is used with cotton, when do you measure the percentage?

J. J. Brown: Fabric stretch for cotton and other type fabrics was measured on 2-inch wide strips of fabric on an Instron Tensile Tester at a 2-pound load.

Percent stretch =

$$\frac{\text{length at 2-lb. load} - \text{relaxed length}}{\text{relaxed length}}$$

Question: When will you be able to estimate costs?

J. J. Brown: From information which we have been able to obtain, it is estimated that the cost of stretch 24/2 cotton yarns would be around $1.25 per pound. Until stretch cotton yarns are produced in commercial plants where workloads, etc. are worked out, $1.25 per pound for 24/2 is the best information we have.

Question: What are the merits of greige U.S. bleached goods in slack mercerizing? Compare chainless U.S. chain mercerizer for slack mercerizing.

W. G. Sloan: In some cases slack mercerization after bleaching may result in a harsher feel or hand. Our experience has been that the gross shrinkage after slack mercerization of bleached goods and grey goods has been approximately the same. However, we have received reports that slack mercerization after bleaching results in less shrinkage.

A chainless mercerizer is designed to prevent filling shrinkage by having the cloth in contact with the surface of the rolls at all times. To obtain filling shrinkage, it is necessary to sky the fabric over the rolls so that shrinkage can take place. A chain mercerizer is satisfactory for slack mercerization by reducing or eliminating washing and running the chain in to the shrunken width.

Question: The usual stretchability for cotton stretch is about 15 to 20 percent. What is the possibility of obtaining a greater percentage of stretch from cotton stretch? Incidentally, there was a newspaper article mentioning 200 percent stretch.

W. G. Sloan: It is possible to obtain more than 15- to 20-percent easy stretch in slack mercerized filling stretch fabrics by making changes in fabric construction such as (1) using fewer warp ends per inch; (2) using smaller filling yarns than warp yarns; (3) using a higher twist in the filling; or (4) by using a weave which has more stretch such as a honeycomb or leno weave. However, as easy stretch is increased, the percent growth usually increases also so that fabrics having in excess of 20 percent easy stretch may have too much growth. It is preferable to have a maximum of 3-percent growth after the static load test.

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